

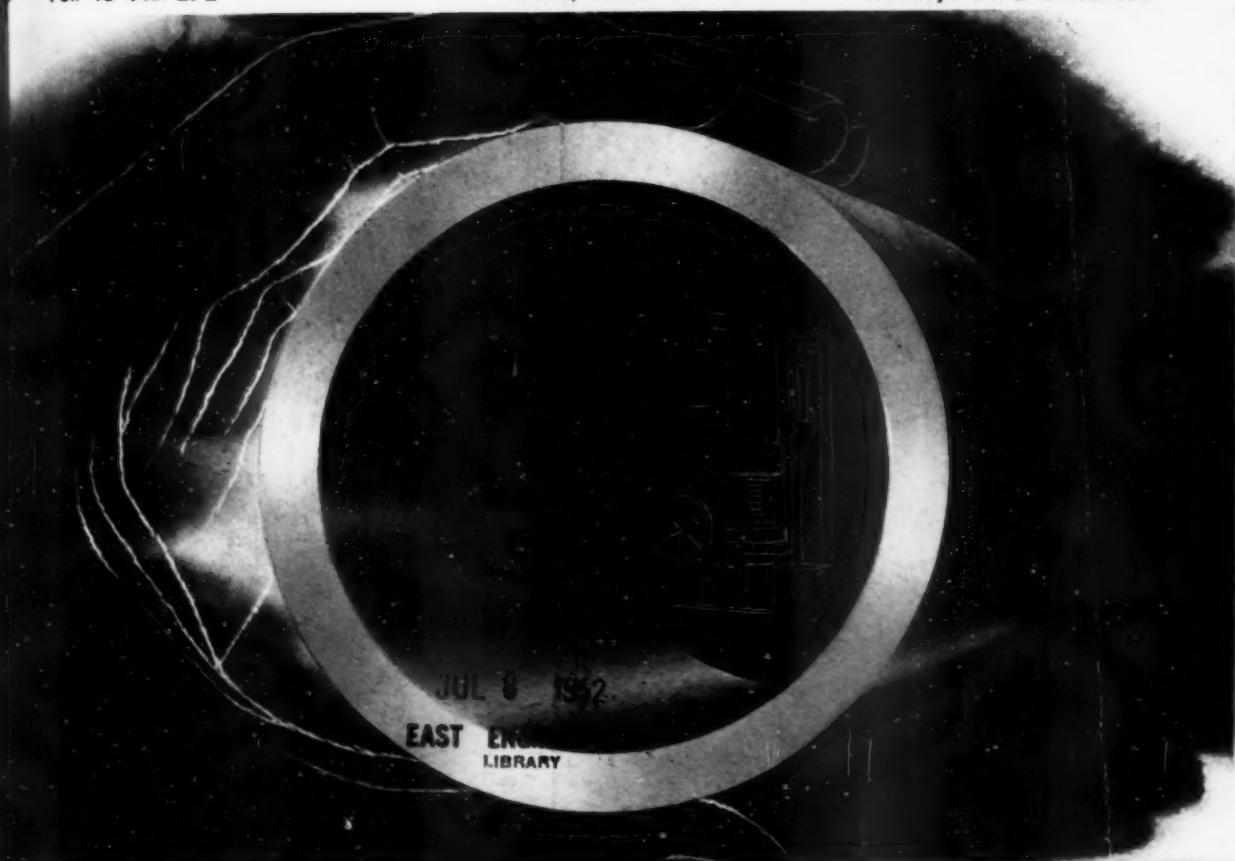
# METALLURGIA

THE BRITISH JOURNAL OF METALS

Vol. 45 No. 272

JUNE, 1952

Monthly: TWO SHILLINGS



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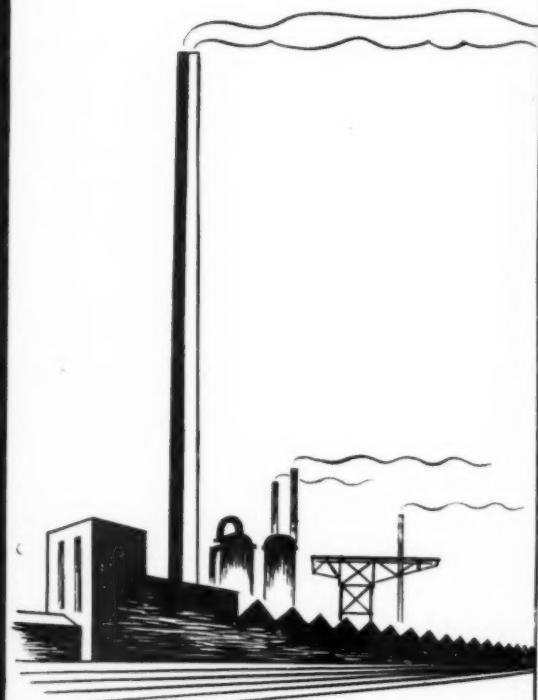
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# METALLURGIA

THE BRITISH JOURNAL OF METALS  
INCORPORATING THE "METALLURGICAL ENGINEER."

JUNE, 1952

Vol. XLV No. 272

## Scientific Man-Power

**I**N opening a debate in the House of Lords, the other day, on the use made by industry and agriculture of the results of scientific research, Lord Samuel said that Britain had been a pioneer in revolutionary scientific discoveries, but America, Switzerland and some other countries had in some respects surpassed us in practical working. The newer industries had a good record of adapting themselves to new methods, but the older industries' record was by no means so good, and it was having the most harmful effects.

In the same debate, Lord Woolton pointed out that the new synthetic fibre Terylene, discovered in Manchester in 1946, was at least as valuable as nylon, but although research and development had been pressed forward with great energy and speed here by I.C.I., Terylene was going into production faster in the United States, and products made from it were already beginning to compete with our own textiles in the export markets of the world. "Here," added Lord Woolton, "it would seem that our main handicap is that the construction of a chemical manufacturing plant takes about twice as long as it did before the war. This may well be due to delays through material shortages, and it is an open question whether it could have been avoided."

Although not perhaps a typical example of the causes underlying delayed application of scientific results—for this is a case where the possibilities were quickly realised and steps taken to put the development on a production scale—it does emphasise most strikingly the harm which can come from this slackness in our industrial make-up. True, our circumstances are such that we cannot afford to rush into production of a new product, or completely revolutionise our methods of manufacture, without adequate investigation. And that leads to the crux of the matter—the shortage of highly trained technologists capable of appreciating the possibilities of the results of a particular piece of scientific research and of translating them into a commercial proposition.

Although the difficulty of equating the respective academic standards and awards of Britain and the United States introduces an element of uncertainty, it is estimated that in 1949-50 the number of people qualifying in science and technology to our Higher National Certificate standard was 110,000 in America, in comparison with 14,000 here. Even allowing for the disparity of the size of the labour force in the two countries, America is turning out, and presumably employing, nearly three times as many scientists as we are in Great Britain. More than that, the Americans are planning to increase their facilities for training scientists, particularly at the higher levels where we at present enjoy a relative advantage. A similar disparity exists between this country and Switzerland, whose

population and resources of indigenous wealth are very much smaller than our own.

In the Fifth Annual Report of the Advisory Council on Scientific Policy (1951-52), published recently by H.M. Stationery Office, the view is expressed that a policy for scientific manpower can in fact proceed on only one of two alternative assumptions. We can either assume that there will be a steady and rapid increase in our productivity, or that the economy will remain static and that productivity will increase only slowly. If we accept the first alternative, we must plan to meet the demand for scientists to which it will give rise, and on which it depends. If the second assumption is correct, there is little hope of our remaining a great power, or even of our paying for the imports needed to sustain our economic life. If planning is to have any purpose it must, therefore, be designed to meet the needs and create the situation of the first hypothesis.

How is the situation to be remedied. It is obvious that the two essentials of any scheme are the raw materials, in the way of trainees, and the means of training them—suitably equipped and staffed institutions, whether technical colleges or universities. One cannot hope for any immediate and dramatic increase in the facilities for the training of higher technologists, but the subject has been under consideration by the Government, and it is considered that such facilities can best be improved by building up at least one institution of university rank devoted primarily to the teaching and study of the various forms of technology. The practical possibilities as to the best way of doing this are, therefore, being urgently explored. At the same time, the Government fully recognises the important contribution which technical colleges could make to higher technological education, and details of proposals for making improved financial assistance available for selected colleges and courses therein are to be announced shortly.

More men possessing the highest qualities of leadership and personality, and at the same time equipped with a scientific training, must be encouraged to the front but as the number of men possessing high qualities of intellect and leadership is limited, a substantial increase in the number of those with such qualities who take up science can only be attained at the expense of other studies. This is unlikely to happen without a change in opinion about the prestige of science for, whilst the situation in the grammar schools—where a high proportion of the best boys take up science—is satisfactory, the same cannot be said of the public schools, where science does not attract as many boys of outstanding ability as is desirable in the national interest.

Finally, there must be a change in attitude in the board-rooms in industry where, in many cases, an understanding of the scientific basis of technical progress is still not regarded as an asset. To compete in world markets we need to offer to first class technologists the necessary salaries, prospects and responsibilities.

# Queen's Birthday Honours

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## New Fellow of Royal Society

UNDER the Statute of the Royal Society which provides for the election of persons who either have rendered conspicuous service to the cause of science or are such that their election would be of signal benefit to the Society, Sir Edward Bridges, G.C.B., G.C.V.O., was elected a Fellow of the Society on May 15th, 1952.

# The Creep and Fatigue Properties of Two Commercial Aluminium Bronzes at 500°C.\*

By J. J. Carter, B.Met.E.,† D. N. Mends, B.Sc.(Eng.), A.I.M.,‡

and J. McKeown, D.Sc., M.I.Mech.E., F.I.M.,§

(Communication from the British Non-Ferrous Metals Research Association)

*This paper is part of a study by the Association, still in progress, of copper-base alloys for use in heat exchanger tubes of gas turbine engines.*

To provide a basis of comparison in the development of copper-base alloys for use in tubular heat exchangers for gas turbines, tests have been made on two alloys at present commercially available and in use. Each of these alloys contains approximately 7% aluminium, the first containing in addition 2% nickel and the second 1% each of iron and nickel and 0.3% manganese. For convenience in this paper the first of these alloys is referred to as "Alloy A" and the second as "Alloy B".

The chief attraction of these materials is their resistance to scaling at the temperatures experienced in existing gas turbine heat exchangers. The alloys are known to form thin, adherent and protective scales when exposed to air at temperatures of the order of 500°C. and opportunity was taken in the present work to record whether or not this behaviour was unchanged when the materials were subject to static and cyclic stresses in air at 500°C.

Several supplies of each of the two materials were subjected to creep testing and the details of the production of each supply are given in the Appendix. The materials were supplied with the same degree of cold working as that used in the production of actual heat exchanger tubes. Throughout the paper the materials in the "as received" condition are referred to as the cold worked materials.

The materials have been tested in both the cold worked and the annealed conditions and the effect of grain size on the creep resistance of the annealed materials has been investigated. In general, the creep and stress-rupture time tests have been made on strip

cut from tubes or from rolled material of the same thickness as the tube wall, while the fatigue tests have been made on specimens machined from 1 in. diameter rod.

As the work progressed it became clear that the materials were not structurally stable at the temperature of test (500°C.), whether they were in the cold worked condition or whether they had been annealed at temperatures in the range 700°-900°C. and air cooled. Accordingly, an account is given of the changes occurring when the alloys are held for long periods at temperatures of 500° to 600°C. without applied stress.

## Materials

*Alloy A*.—Three separate supplies of this material were obtained, the first two in the form of square-section drawn tubes, 1½ in. × 1½ in. × 0.064 in. wall thickness, and the third as drawn rod 1 in. diameter.

*Alloy B*.—Three separate supplies of this alloy were also obtained, the first as drawn tube to the same dimensions as for Alloy A, the second as drawn rod 1/16 in. diameter and the third as rolled strip produced in the Association's laboratories.

The creep and stress-rupture time specimens were cut longitudinally from the tubes and rolled strip and the fatigue specimens from the rod. Some creep specimens machined from the rod were also tested. The production details of the various materials are given in the Appendix, while chemical and spectrographic analysis results are given in Table I.

## COLD WORKED MATERIALS

Room temperature tensile and hardness properties of the commercial materials in the cold worked condition are given in Table II. The metallographic structures of these materials were as follows:—

*Alloy A*.—The structures consisted of heavily twinned alpha grains, showing some evidence of the direction of

\* B.N.F.M.R.A. Report. R.R.A. 928P. The work described in this paper was made available to members of the British Non-Ferrous Metals Research Association in confidential research reports issued in 1950 and 1951.

† Formerly Bursar, B.N.F.M.R.A., London.

‡ Formerly Investigator, B.N.F.M.R.A.

§ Head of Mechanical Testing Section, B.N.F.M.R.A., London.

TABLE I.—CHEMICAL AND SPECTROGRAPHIC ANALYSES.

Element	Alloy A			Alloy B			Approx. Limit of Spectrographic measurement
	Tube	Tube	Rod	Tube	Rod	Rolled Strip	
Chemical	%	%	%	%	%	%	%
Aluminium	7.01	6.71	6.96	7.07	6.48	7.05	
Iron	0.06	0.06	0.03-0.05	0.83	0.90	0.86	
Nickel	2.19	2.17	2.07	0.89	0.71	0.98	
Manganese	0.04	0.01	0.01	0.30	0.31	0.02	
Spectrographic							
Bismuth	<0.001	<0.001	n.d.	n.d.	n.d.	n.d.	0.001
Lead	0.003	<0.001	n.d.	0.003	<0.001	n.d.	0.001
Tin	0.01	n.d.	n.d.	n.d.	n.d.	n.d.	0.01
Silicon	<0.02	<0.03	<0.03	0.03	<0.02	<0.01	0.01
Arsenic	n.d.	<0.02	0.005	n.d.	n.d.	n.d.	0.003
Zinc	<0.05	n.d.	trace	<0.01	<0.02	n.d.	0.01

n.d. = not detected spectrographically.

TABLE II.—ROOM TEMPERATURE PROPERTIES AND GRAIN SIZES OF COLD WORKED MATERIALS.

	Alloy A		Alloy B	
	Tube	Rod	Tube	Rod
0.1% Proof Stress—tons/sq. in. . . . .	22.3	25.0	25.2	25.2
Tensile Strength—tons/sq. in. . . . .	33.9	35.5	36.6	40.2
Total Elongation % . . . . .	29.3 on 2 in.	43.5 on $4\sqrt{A}$	43.0 on 2 in.	35.5 on $4\sqrt{A}$
Hardness—V.P.N. . . . .	160–190	180–205	175	160–170
Grain Diameter—mm. . . . .	0.06	$\leq 0.06$ (V)	0.10	$\leq 0.10$ (V)

V = Variable.

cold working, particularly in the case of the rod. The grain size of the tubes was the same in the two supplies, but that of the rod was much finer and too variable to permit proper measurement. (See Table II.)

**Alloy B.**—The structure of the tubes was similar to that of the Alloy A tubes but with a coarser grain size, and the structure of the rod was similar to that of the Alloy A rod.

#### ANNEALED MATERIALS

To obtain materials in the fully annealed condition and also to obtain in the materials a number of different grain sizes, with a view to examining the influence of this variable on creep properties, various heat treatments were used, each treatment being followed by air cooling.

When the Alloy A tubes were annealed for 2 hours at 695° C. they showed a fully recrystallised single phase structure with extensive twinning, and a reasonably uniform grain diameter of 0.037 mm. Annealing for 2 hours at 750° C. gave a similar structure and a grain diameter of 0.082 mm. When rod specimens of this alloy were annealed for 2 hours at either 750° C. or 900° C., fully recrystallised structures were obtained, the grain diameters being 0.22 mm. and 0.5 mm. respectively. With both treatments there was a small amount of grain boundary precipitate in the form of dark spots.

The Alloy B tubes did not recrystallise after annealing for up to 24 hours at 700° C. but after annealing for 24 hours at 750° C. a fully recrystallised structure was obtained, the grain diameter being 0.037 mm., i.e., the same as that of Alloy A tubes annealed at 695° C. The Alloy B rod was not completely recrystallised after 24 hours at 750° C. The other treatments, which produced complete recrystallisation, and the grain diameters obtained were:—

½ hour at 850° C.—0.12 mm.  
2 hours at 900° C.—0.23 mm.

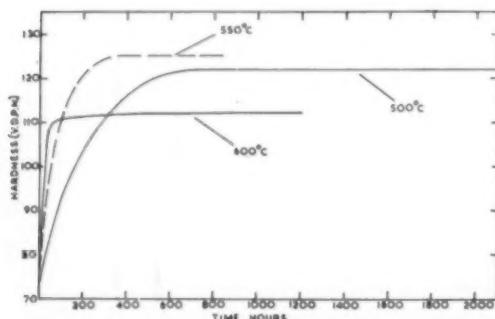


Fig. 1.—Age hardening curves for Alloy A. Air cooled from 750° C. and aged at the temperatures shown.

Specimens of this alloy with other grain sizes were obtained by taking some of the hot rolled material produced in the laboratory, cold rolling it 20% reduction and annealing either 1½ minutes at 725° C. or ½ hour at 860° C., these treatments resulting in grain diameters of 0.091 mm. and 0.30 mm. respectively. No precipitate, either within the grains or at the grain boundaries, was detected in any of the samples of annealed Alloy B.

The room temperature properties and grain diameters of the annealed materials are given in Table III.

#### THE EFFECT OF AGEING TREATMENTS ON THE HARDNESS AND STRUCTURES OF THE ANNEALED MATERIALS

Hardness changes were observed in specimens of both materials following creep testing at 500° C. The changes occurring in specimens which were tested in the cold worked condition could be attributed in part to recrystallisation, either partial or full, but changes which occurred in the annealed specimens, particularly in Alloy A, indicated that other structural changes were occurring, and these were explored further.

In tubular heat exchangers for gas turbines, the tubes are of the order  $\frac{1}{2}$  in. diameter and 22 s.w.g. wall thickness and may be 20 feet long. Such tubes could not conveniently be quenched from a high solution-treatment

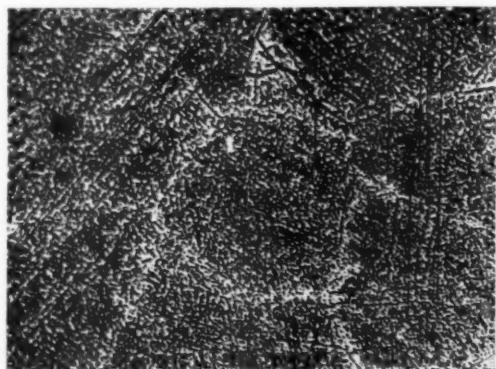


Fig. 2.—Alloy A after 336 hours stress-free ageing at 500° C., following air cooling from 750° C.  $\times 250$

temperature. This factor has been borne in mind in the investigation of the effects of ageing treatments on the mechanical properties of the alloys, and the more practicable procedure of air cooling has been used. Ageing tests have been carried out, on samples of rod, to determine changes in hardness and structure resulting from holding at 500° C. and, with Alloy A, at 550° and 600° C., all after air cooling from higher annealing temperatures.

**Alloy A.**—After air cooling from 750° C. the specimens were held at the ageing temperature for periods up to 2,350 hours and air cooled. The changes in hardness with time are shown in Fig. 1. Maximum hardness was reached in 670 hours at 500° C., and no further change occurred in the next 1,680 hours.

After 6 hours at 500° C., the amount of the grain boundary precipitate previously noted in the annealed specimens had increased only slightly. After 25 hours, the first visible precipitate within the grains had appeared as dark spots. These increased in size and number, and after 96 hours had assumed a clearly acicular shape. Fig. 2 shows the structure after 336

hours. At this stage, a set of "ghost" grain boundaries had appeared, distinct from the real grain boundaries; they were light in colour, representing regions of no precipitation, and were the remains of a previous structure. It is possible that precipitation had occurred at grain boundaries at some stage during the fabrication of the rod. Subsequent heat treatment, either during or after fabrication, moved the position of the grain boundaries, without being sufficient to produce complete homogenisation.

Between 336 and 2,350 hours the precipitated particles became gradually larger, and in a few places began to form agglomerates as shown in Fig. 3. At the grain boundaries, the precipitate formed globular particles, and there were areas without any precipitate near some of the grain boundaries.

*Alloy B.*—The specimens were annealed for  $\frac{1}{2}$  hour at 850° C., cooled in still air, and thereafter aged at 500° C. for periods up to 840 hours. The age-hardening curve appeared to show two maxima, the first being reached after 24 hours and the second after 672 hours. However, the greatest increase in hardness measured during this time was only 6 V.P.N. After 72 hours ageing, dark spots appeared in the grain boundaries (Fig. 4). This

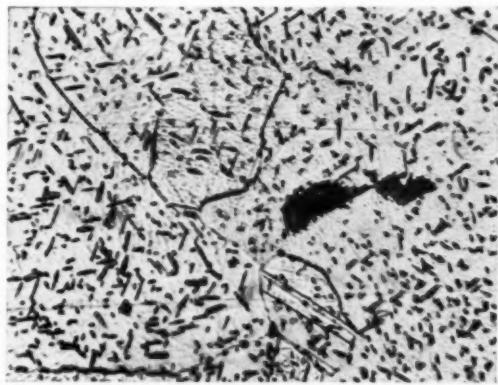


Fig. 3.—Alloy A after 2,350 hours stress-free ageing at 500° C., following air cooling from 750° C.  $\times 1,000$

precipitation may have been the cause of the first maximum in the age-hardening curve. The grain boundary particles increased in size and number during the next 768 hours. At 672 hours the first visible precipitate appeared within the grains. In places the particles were acicular in form, similar to those observed during the ageing of Alloy A.

#### Creep and Stress-Rupture Time Tests

##### EQUIPMENT AND TEST METHODS

The creep and stress-rupture time tests were carried out on creep units of B.N.F. design which are fully described elsewhere<sup>1</sup>. Two types of specimens were used, (i) a flat specimen machined from the tubes or the rolled strip, having a width of 0.5 in. on a 5 in. parallel length and an effective gauge length of 6 in., (ii) a round specimen machined from the rods, having a diameter of 0.357 in. on a 5 in. parallel length and an effective gauge length of 5.47 in. In both types of test the temperature of the specimen was thermostatically controlled to

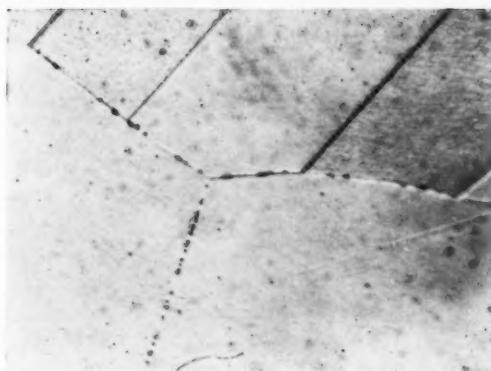


Fig. 4.—Alloy B after 72 hours stress-free ageing at 500° C., following air cooling from 850° C.  $\times 1,500$

$\pm 1^\circ$  C. and the uniformity of temperature along the parallel length was within  $\pm 1.5^\circ$  C.

The specimens were raised to the test temperature in about 4 hours and held at that temperature for a further 20 hours before the load was applied. In the creep tests the extension of the specimen was measured on two sides by micrometers which were read to 0.0001 in., i.e., the strain was measured to approximately  $2 \times 10^{-5}$ . (Actually  $1.67 \times 10^{-5}$  in the 6 in. gauge length specimen and  $1.82 \times 10^{-5}$  in the 5.47 in. gauge length specimen.) In the stress-rupture time tests the times to fracture were measured by electric clocks having trip switches actuated by the fracture of the specimen.

##### CREEP TESTS ON COLD WORKED MATERIALS

*Alloy A.*—The two supplies of tube, although having the same grain size, gave somewhat different creep curves, the second supply being slightly less creep resistant than the first supply at 4,000 and 5,000 lb./sq. in. and slightly more creep resistant at 3,000 lb./sq. in. At each stress a complete three-stage creep curve was obtained for plastic strains less than 1%, the tertiary stage commencing at about 0.5% strain. The relationship between stress and time-to-produce a number of specific values of plastic strain is given in Fig. 5.

TABLE III.—ROOM TEMPERATURE PROPERTIES AND GRAIN SIZES OF ANNEALED MATERIALS.

	Alloy A			Alloy B		
	Tube	Tube	Rod	Tube	Rod	Rolled Strip
Annealing Treatment	2 hours at 695° C.	2 hours at 750° C.	2 hours at 750° C.	24 hours at 750° C.	1 hour at 850° C.	1½ min. at 725° C.
0.1% Proof Stress—tons/sq. in.	9.1	5.7	7.4	9.0	9.4	7.2
Tensile Strength—tons/sq. in.	27.3	27.6	28.8	29.2	29.2	27.3
Elongation %	on 2 in. 65	on 2 in. 65	on 4√A 60	on 2 in. 57	on 4√A 55	on 2 in. 63
General† . . .	8	6	19	7	20	11
Local . . .						
Hardness—V.P.N.	98	71	71	92	77	71
Mean Grain Diameter—mm.	0.037	0.082	0.22	0.037	0.12	0.091

\* Flash anneal in salt bath.

† The "general" extension is that which occurs in the tensile test up to the point of maximum load. The extension which occurs between the maximum load and fracture is the "local" extension and corresponds to the stage of necking of the specimen. The total extension is the sum of the general and local extensions.

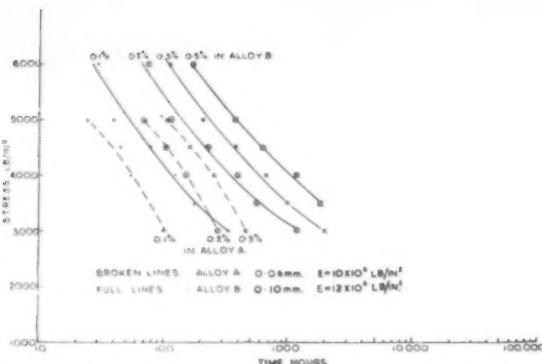


Fig. 5.—Stress-time for specific creep strains in cold worked tube materials.

The rod material with a much smaller grain size than that of the tubes, was much less resistant to creep at stresses of 3,000 and 4,000 lb./sq. in., its creep strain at 200 hours being double, and at 800 hours six times, that of the tubes.

*Alloy B.*—The tube material was tested over a range of stresses from 3,000 to 6,000 lb./sq. in. This material had a somewhat larger grain size than the Alloy A tubes and its resistance to creep was greater than that of Alloy A. The relationship between stress and time to produce a number of specific values of plastic strain is given in Fig. 5.

The rod material, with a much finer grain size, had a much lower creep resistance than the tube. Thus at 3,000 lb./sq. in. the creep strain of the rod at 200 hours was four times, and at 500 hours eight times, that of the tube.

#### STRESS-RUPTURE TIME TESTS ON COLD WORKED MATERIALS

The results of these tests, made on specimens from tubes, are given in Fig. 6 in which, for the sake of clarity, the times to failure for only the second supply of the Alloy A tubes are included. The first supply gave longer times to failure at high stresses, 28 hours compared with 7 hours at 10,000 lb./sq. in., and shorter times, 1,400 hours compared with 3,800 hours, at low stresses. The times to failure were greater for Alloy B than for

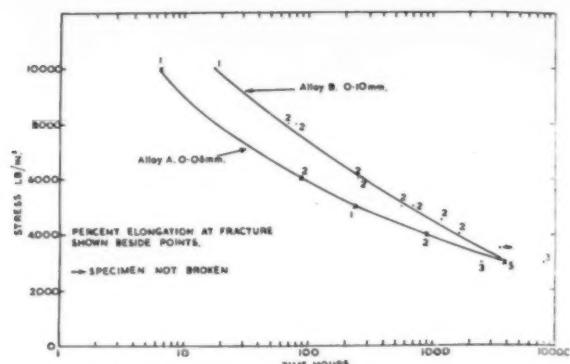


Fig. 6.—Stress-rupture time tests on cold worked materials.

Alloy A at the high stresses but the difference decreased at lower stresses and the materials gave the same time to failure (4,000 hours) at a stress of 3,000 lb./sq. in. In both alloys there is an indication that the elongation at fracture is increasing with increase of life. As shown later, in the section on metallographic examination after test, slow recrystallisation occurred during the tests and this may have been responsible for the increasing ductility with increasing life.

#### STRUCTURES AFTER CREEP AND STRESS-RUPTURE TIME TESTS OF COLD WORKED MATERIALS

*Alloy A.*—Partial recrystallisation had occurred in all specimens which had been on test for a time greater than 200 hours. In those specimens which fractured, the fracture was intercrysalline, and in those in which recrystallisation had occurred the fracture followed the new grain boundaries (Fig. 7).

Both discontinuous and continuous precipitation\* were detected in specimens which failed after 6½ hours under stress (26½ hours at temperature) and in specimens which failed in longer times the amount of both types of precipitation was greater. Intercrysalline cracks

\* The term "discontinuous precipitation" is generally used to describe the breakdown of a supersaturated solid solution by the concurrent separation of the new solid solution and the second phase, which are in (or near) equilibrium at the ageing temperature. The term "continuous precipitation" is used to describe the alternative mode of breakdown, in which the old solid solution is progressively impoverished in the solute with the concurrent separation of the equilibrium precipitate.

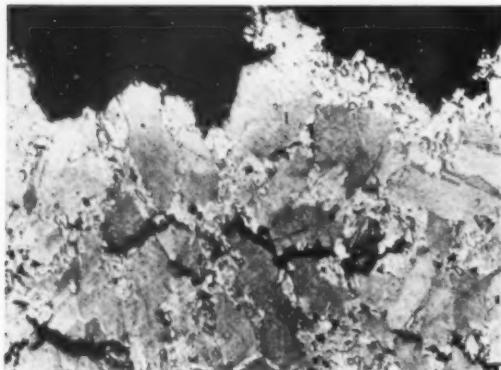


Fig. 7.—Alloy A. Creep fracture in cold worked tube material. Stress: 4,500 lb./sq.in.; life: 520 hours; elongation: 2%.

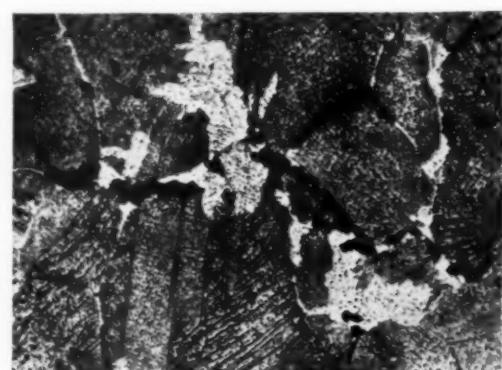


Fig. 8.—Alloy A. Structure in cold worked tube specimen, after fracture. Stress: 4,000 lb./sq.in.; life: 900 hours; elongation: 2%.

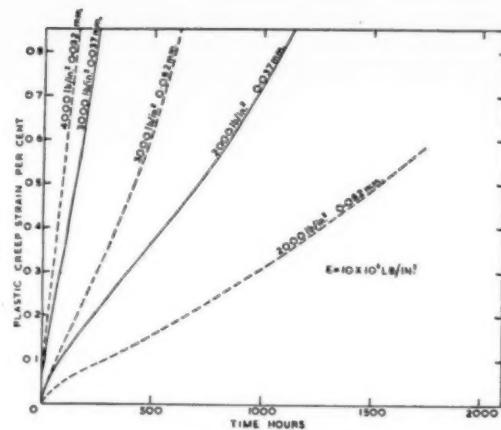


Fig. 9.—Creep tests on annealed Alloy A tube.

which formed approximately at right angles to the direction of stress were in some places associated with the discontinuous precipitation (Fig. 8), but it cannot be stated definitely that these phenomena were related. Grey globular particles were formed during discontinuous precipitation. In the tube material, the regions in which discontinuous precipitation had occurred after 900 hours at 4,000 lb./sq. in. covered about 10% of the field of view. In the rod material, they covered about 70% of the field after testing for 650 hours at 3,000 lb./sq. in. The continuous precipitation within the original grains was mainly globular but sometimes acicular, as in the precipitate associated with stress-free ageing.

*Alloy B.*—No recrystallisation had occurred in the rod specimens in times up to 650 hours. In the case of the tube material, partial recrystallisation was observed only in those specimens which had been at the test temperature for longer than 1,600 hours. Structural changes were observed similar to those occurring after stress-free ageing of the annealed material for similar periods of time. All fractures were intercristalline.

#### CREEP TESTS ON ANNEALED MATERIALS

*Alloy A.*—The creep curves for this alloy with two different grain sizes are given in Fig. 9 and show that the material with the larger grain size had the greater resistance to creep. The relationship between stress and

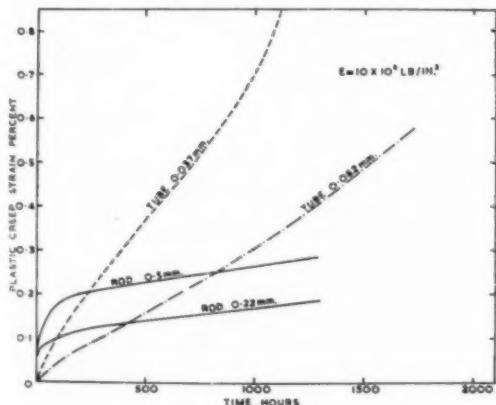


Fig. 11.—Creep tests at 2,000 lb./sq.in. on annealed Alloy A in various grain sizes.

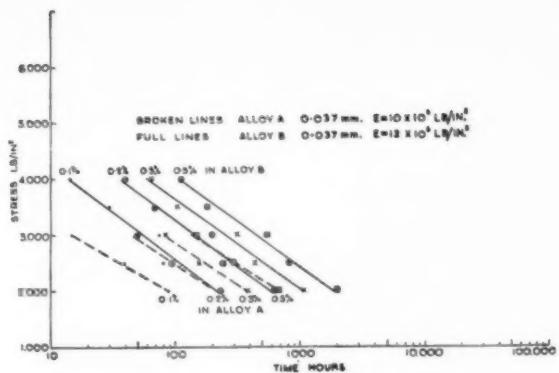


Fig. 10.—Stress-time for specific creep strains in annealed tube materials.

time for a number of specific values of plastic strain is shown in Fig. 10 for the finer grained material.

The rod material was annealed for 2 hours at 750° C. giving a grain diameter of 0.22 mm., and also for 2 hours at 900° C. the grain diameter being 0.50 mm. These specimens were tested at 2,000 lb./sq. in. and Fig. 11 shows the creep curves obtained together with those of annealed tube specimens of two other grain sizes. The effect of grain size on the rate of secondary creep is seen to be quite pronounced for grain diameters between 0.037 and 0.22 mm. Further increase in grain size produced little further change in rate of secondary creep but a pronounced change in the extent of primary creep.

Fig. 12 shows that the annealed material had a lower resistance to creep than the cold worked material, even when it had a larger grain size.

*Alloy B.*—The creep curves for specimens of this material machined from tube and having a grain diameter of 0.037 mm. are shown in Fig. 13, while the relationship between stress and time to produce a number of specific values of plastic strain is shown in Fig. 10. An additional creep curve at 2,000 lb./sq. in. is given in Fig. 13 for a specimen of rod annealed to have a grain diameter of 0.23 mm. and shows the considerable effect on creep resistance of this increase in grain size.

Further tests on the effect of grain size on the creep behaviour of annealed Alloy B were made on specimens

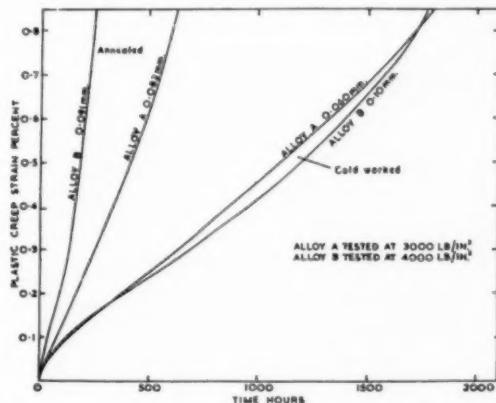


Fig. 12.—Comparison of cold worked and annealed materials.

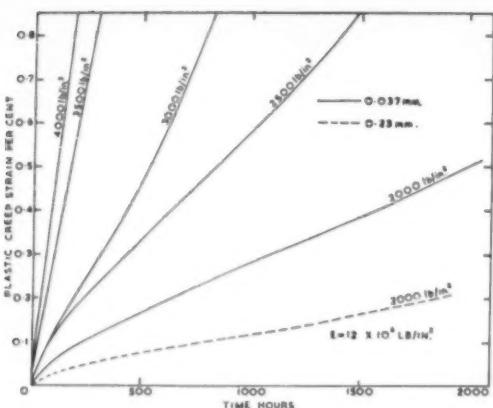


Fig. 13.—Creep tests on annealed Alloy B.

of the rolled strip annealed to give grain diameters of 0.091 mm. and 0.30 mm. These were tested at 4,000 lb./sq. in. and the data obtained from these and a specimen of the tube material tested at the same stress are as follows :

Grain Diameter mm.	Plastic Creep Strain % at 200 hours	Time to Fracture hours	Extension at Fracture %
0.037	0.94	380	7
0.091	0.58	280	12
0.30	0.085	250	<1

It is seen that while an increase in grain size increased resistance to creep it did not increase the life of the specimen, increased grain size being coupled with reduced ductility at fracture.

The creep curves given in Fig. 12 show that Alloy B is more resistant to creep in the cold worked than in the annealed condition, when grain size is similar in the two states. The comparison in Fig. 10 of the two alloys in the annealed condition and at the same grain size shows that Alloy B is basically more resistant to creep than Alloy A.

#### STRESS-RUPTURE TIME TESTS ON ANNEALED MATERIALS

The results of these tests are given in Fig. 14, in which two curves are given for Alloy A in two different grain sizes. These two curves run approximately parallel for times up to 1,000 hours, the longer lives being given by the coarser grained material. A direct comparison can be made between the two alloys at the

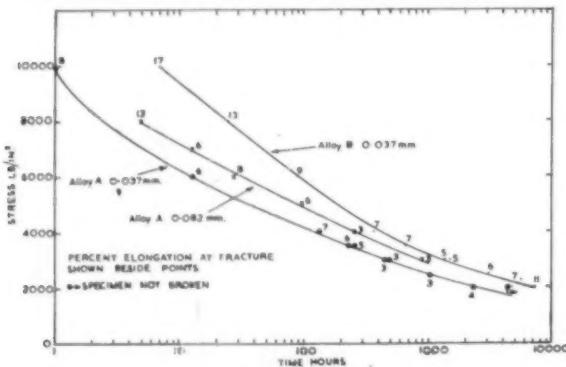


Fig. 14.—Stress-rupture time tests on annealed materials.

same grain diameter, 0.037 mm., and shows that Alloy B has the longer life at all stresses. In the Alloy A at both grain sizes the ductility at fracture decreased as the life increased. This also occurred in Alloy B for times up to about 2,000 hours but the ductility increased again at longer lives.

#### STRUCTURES AFTER CREEP AND STRESS-RUPTURE TIME TESTS ON ANNEALED MATERIALS

*Alloy A.*—In the creep specimens, continuous precipitation within the grains was extensive and acicular in form. Globular particles were present in the grain boundaries, and regions adjacent to some of the grain boundaries were free from precipitate (Fig. 15). Structures in those stress-rupture time test specimens which failed after long times were similar to those of the creep specimens. Specimens which failed in less than 100 hours showed discontinuous precipitation at some of the grain boundaries, as well as a small amount of continuous precipitation both within the grains and in some grain boundaries. All fractures were intercrystalline.

*Alloy B.*—The only structural changes observed were similar to those which occurred during stress-free ageing for similar periods of time. All fractures were intercrystalline.

#### Fatigue Tests

These tests were carried out on each alloy in the cold worked and in the fully annealed conditions, the specimens being machined from the drawn rod. The tests were made on B.N.F. Rotating Load Fatigue Testing machines described elsewhere<sup>2</sup>. Specimens were brought up to the test temperature in 4 hours and held for a further 20 hours before stressing. The temperature, thermostatically controlled to  $\pm 2^\circ \text{C}$ , was measured by a thermocouple attached to the specimen with asbestos tape at the position of maximum stress. The surface of the specimen in the region of maximum stress was polished longitudinally using 00 emery paper and the specimens degreased before placing in the machine. The speed of testing was 3,000 cycles/minute.

#### COLD WORKED MATERIALS

Considerable scatter of results was obtained in the tests on both materials and it is thought that this was due to the lack of uniformity of cold working in the rod, as shown by hardness tests (see Table II).

*Alloy A.*—The endurance curve is shown in Fig. 16 as a band. The band is still falling at  $30 \times 10^6$  cycles, and this is undoubtedly due in part to oxidation of the specimen surface increasing with increased time of test. The bending stress being a maximum on the surface, the extent of oxidation has a considerable effect on the endurance at any stress. In other words, the tests were "oxidation-fatigue" tests, and some of the scatter of results may have been due to variation in oxidation of the specimen surface. In some cases the scale present in the region of maximum stress was exfoliative, in contrast to the adherent scale formed during stress-free ageing and the similar scaling behaviour under static stress in the creep tests.

*Alloy B.*—The endurance band is shown in Fig. 17. It is seen to be still falling at  $100 \times 10^6$  cycles, probably because of continuing oxidation of the specimen surface.

<sup>2</sup> J. McKeown and J. H. Back. *Metallurgia*, 1948, **38**, 247-254.

The endurance limit of both materials at  $50 \times 10^6$  cycles appeared to be of the order of  $\pm 10$  to  $\pm 11$  tons/sq. in.

#### STRUCTURES AFTER TESTS ON COLD WORKED MATERIALS

Transverse and longitudinal sections near each fracture were examined, the longitudinal sections being taken through the fracture surface in the region considered to include the start of the fracture. No inclusions which might have adversely affected the endurance were detected. In some cases, intercrystalline surface cracking was detected in the vicinity of the fracture, but the greater portion of each fracture was transcrystalline. No recrystallisation had occurred during any test. In the Alloy A specimens continuous and discontinuous precipitation of the same nature as that observed after creep tests on this material had occurred to about the same extent as in the creep specimens tested for similar times. In the Alloy B specimens no precipitation of any kind was detected.

#### ANNEALED MATERIALS

The tests on Alloy A were made on specimens annealed for 2 hours at  $750^{\circ}\text{C}$ . and the endurance curve is shown in Fig. 16. The specimens of Alloy B were annealed for

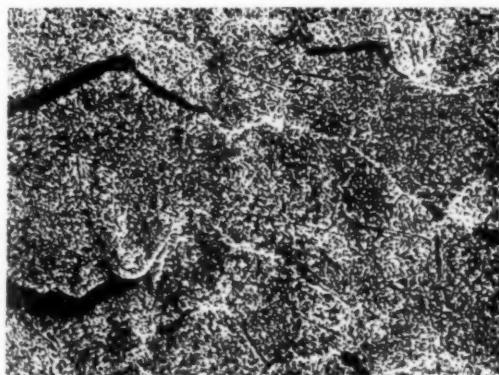


Fig. 15.—Alloy A. Structure in annealed tube creep specimen, after fracture. Stress: 3,000 lb./sq.in.; life: 880 hours; elongation: 3%.  $\times 250$

½ hour at  $850^{\circ}\text{C}$ . and the endurance curve is shown in Fig. 17. In both cases the curves are seen to be falling steeply even at  $50 \times 10^6$  cycles, probably because of the oxidation effect referred to above. The endurance limit of both materials at  $50 \times 10^6$  cycles is of the order of  $\pm 5$  to  $\pm 6$  tons/sq. in.

#### STRUCTURES AFTER TESTS ON ANNEALED MATERIALS

The fractures were mainly transcrystalline, with some intercrystalline cracking at the surface near the start of the fracture. There was considerable deformation in the region of a fracture, and even several grains away from the fracture surface. In Alloy A continuous precipitation only was observed, the degree being about the same as that occurring in equal periods of time in stress-free specimens aged at  $500^{\circ}\text{C}$ . In Alloy B dark spots were present in the grain boundaries similar to those observed in stress-free specimens aged at  $500^{\circ}\text{C}$ . and appearing after approximately the same time (see Fig. 4).

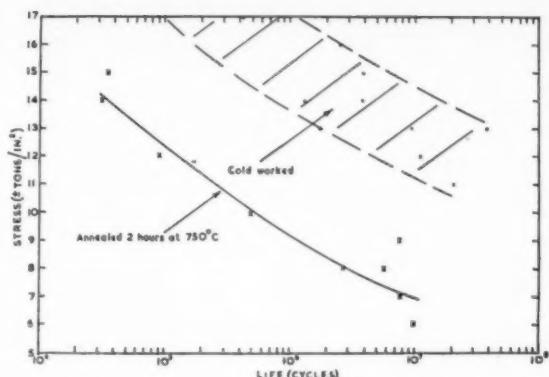


Fig. 16.—Fatigue tests on Alloy A.

#### Summary and Conclusions

The creep and fatigue properties of two aluminium bronzes containing (a) 7% aluminium, 2% nickel (Alloy A) and (b) 7% aluminium, 1% nickel, 1% iron, 0.3% manganese (Alloy B) have been determined at  $500^{\circ}\text{C}$ . and the main features of the results are as follows:—

(1) The materials were obtained in the cold worked conditions (approximately 20% to 30% cold reduction) and recrystallisation occurred during some of the creep and stress-rupture time tests. Recrystallisation occurred more readily in Alloy A than in Alloy B. Thus in the stress-rupture time tests it was detected in specimens of Alloy A in which the life to fracture was of the order of 200 hours or greater, whereas in Alloy B it was detected only in those specimens with a life of 1,600 hours or greater.

(2) Both materials underwent structural changes on holding at  $500^{\circ}\text{C}$ . in either stress-free ageing tests or in creep tests after air-cooling from higher temperatures of the order of  $750^{\circ}$  to  $850^{\circ}\text{C}$ .

In Alloy A pronounced age-hardening was found in specimens held free of stress at  $500^{\circ}$ ,  $550^{\circ}$  and  $600^{\circ}\text{C}$ . after annealing at  $750^{\circ}\text{C}$ . (Fig. 1). Continuous precipitation occurred, being visible firstly as dark particles, which at longer times became mainly acicular in shape (Figs. 2 and 3). When deformation was present, as in the cold worked material, holding at  $500^{\circ}\text{C}$ . produced discontinuous precipitation at the grain boundaries (Fig. 8). When the deformation occurred as creep strain in a test on the annealed material, discontinuous pre-

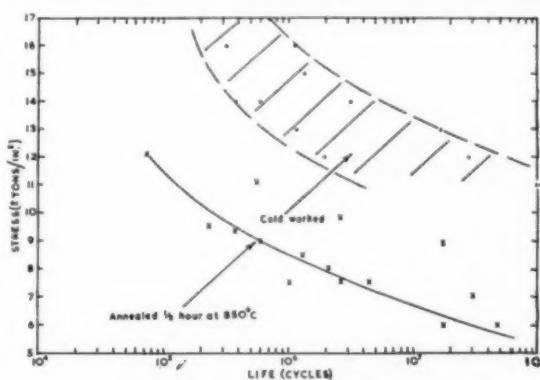


Fig. 17.—Fatigue tests on Alloy B.

cipitation at the grain boundaries occurred in short times of testing, while in longer times only continuous precipitation was observed (Fig. 15).

In Alloy B only slight age-hardening was found in specimens held free of stress at 500° C. after annealing at 850° C. Some precipitate appeared at the grain boundaries after 72 hours (Fig. 4), while after 672 hours precipitate was just visible within the grains. Similar changes were observed in the creep specimens of the cold worked and of the annealed materials in similar times at this temperature.

(3) The tests have shown that the creep and fatigue properties of the cold worked materials are superior to those of the annealed materials but this result is based only on tests of 2,000 to 6,000 hours duration. At the test temperature the cold worked materials were reverting to the annealed condition and hence where much longer service lives than these are envisaged the properties of the annealed materials are the best guide to the useful service properties.

(4) The creep properties of both materials are sensitive to grain size and this probably accounts in large part for the observed differences in the properties of the different supplies of each material tested. The following results illustrate the point: they refer to tests on the annealed materials only and at a stress of 2000 lb./sq. in.

	Grain Diameter mm.	Creep Strain % in:	
		500 hours	1,000 hours
Alloy A	0.037	0.37	0.73
	0.082	0.15	0.31
	0.22	0.14	0.17
Alloy B	0.037	0.17	0.28
	0.23	0.08	0.12

(5) In the cold worked condition the creep resistance of Alloy B was greater than that of Alloy A (Fig. 5). The grain diameter of the Alloy B was 0.10 mm., that of the Alloy A 0.06 mm. and this difference may account in part for the superior creep resistance of the Alloy B. However, Alloy B was also the more creep resistant of the two alloys when both materials were annealed and had the same grain diameter, 0.037 mm. (Fig. 10). Another comparison at a different grain diameter, *i.e.*, 0.22-0.23 mm., is found in the table given under (4) above.

Comparison of the stress-rupture time properties of the two materials is made in Figs. 6 and 14. The properties of the cold worked materials became very similar to those of the annealed materials at times in excess of 5,000 hours, a point already mentioned in (3) above. At shorter times the properties of annealed Alloy B were superior to those of annealed Alloy A but the difference decreased as the time to fracture increased, and it would appear that in times of the order of 10,000 hours or greater there is little to choose between the two alloys. Thus the downward slope of the stress-life curve of Alloy A is less steep than for Alloy B and it is possible that the observed age-hardening of the Alloy A (in times of the order of 500 hours at 500° C.—see Fig. 1) is responsible for this difference in the behaviour of the two materials.

(6) The ductility at failure under creep conditions of annealed Alloy A, especially in those specimens which gave long lives, was somewhat lower than that of annealed Alloy B. Thus at a life of 2,000 hours the elongation at fracture was 4% in Alloy A and 6% in Alloy B.

(7) In rotating bending fatigue tests at 500° C. both alloys gave considerable scatter of results when tested in the cold worked condition and no significant difference was detected between the endurance of the two materials.

When tested in the annealed condition both materials gave more consistent results. Again there was little difference in the endurances of the two materials. A feature of the results obtained was that the endurance curves were still falling noticeably at an endurance of  $50 \times 10^6$  cycles, the stress being between  $\pm 5$  and  $\pm 6$  tons/sq. in. It is considered that this is in part due to continued oxidation of the surfaces of the specimens where the bending stress is a maximum. The tests were, in fact, oxidation-fatigue tests and in this sense may be compared with corrosion-fatigue tests, a well-known feature of which is the continuing fall of the endurance curve with increase in endurance, *i.e.*, increase in time of exposure to the corrosive conditions.

(8) The scaling behaviour of the alloys in air under cyclic stresses at 500° C. was significantly different from their behaviour when the stress was steady or when no stress was applied. Under the latter conditions the scale was always thin and strongly adherent after times up to at least 2,000 hours at 500° C., whereas under cyclic stresses the scale was exfoliative after times as short as 50 hours at the same temperature.

#### Acknowledgments

The authors are indebted to the Director and Council of the British Non-Ferrous Metals Research Association for permission to publish this paper.

#### Appendix: Production Details

In this Appendix are given brief details of the method of manufacture of each raw material used.

##### ALLOY A

1<sup>st</sup> Supply (Square-section drawn tube, 1½ in.  $\times$  1½ in.  $\times$  0.064 in. wall thickness). Round tube 2 in.  $\times$  13 s.w.g. was annealed at 750° C., expanded to 1.985 in. bore  $\times$  0.083 in. thick, plug drawn 1.985 in. bore  $\times$  0.070 in. thick, annealed at 750° C. for ½ hour, sunk shape through penultimate die, plug drawn to 1½ in. square  $\times$  0.064 in. thick, annealed at 450° C. for 1 hour.

2<sup>nd</sup> Supply. Tube produced as for 1<sup>st</sup> supply.

3<sup>rd</sup> Supply (1 in. diameter drawn rod). Cast slugs 6½ in. diameter were extruded at 950° C. to 1½ in. diameter rods. The rods were drawn, in seven stages, to 1.325 in. diameter, annealed 2 hours at 750° C. and air cooled, drawn, in four stages, to 1.192 in. diameter, annealed 2 hours at 750° C. The final reduction to 1.00 in. diameter (29.7% reduction in area) was effected in three stages, the rods being finally reeled to straighten them.

##### ALLOY B

1<sup>st</sup> Supply (Square section drawn tubes, 1½ in.  $\times$  1½ in.  $\times$  0.064 in. wall thickness). Tubes were drawn down to 2½ in. bore  $\times$  0.07 in. thick having been given approximately 30% reduction in area/pass with intermediate anneals at 750° C. A 2½ in. bore tube was annealed at this temperature, roughly squared without any reduction in thickness to approximately 1½ in. square and then finally drawn to 1½ in. outside square  $\times$  16 s.w.g. without further annealing. The finished tube was then low temperature annealed at 450° C. for 1 hour.

2<sup>nd</sup> Supply (1½ in. diameter drawn rod). 8 in. diameter bloom was extruded at 850°-880° C. to 1.22 in. diameter

rod, reeled and annealed at 450° C. for 20 minutes, cold drawn to 1.14 in. diameter, reeled and annealed at 300° C. for  $\frac{1}{2}$  hour, re-annealed at 725° C. for 1 hour, cold drawn to  $\frac{1}{8}$  in. diameter, annealed at 300° C. for 1 hour, heat-treated overnight at 450° C.

3rd Supply (Rolled strip). This material was produced in the Association's laboratories, being made up to the

same nominal composition as the commercially produced Alloy B. From a chill cast ingot 1 in. thick bars were machined  $\frac{1}{2}$  in. thick and 1 $\frac{1}{2}$  in. wide. These bars were hot-rolled from 900° C. in four stages with intermediate and final reheats of  $\frac{1}{2}$  hour at 900° C. The thickness after hot-rolling was 0.1 in., which allowed for subsequent cold-rolling in the treatment used for grain size control.

## Floris Osmond

### A Pioneer in Physical Metallurgy

By Eric N. Simons

*The widespread application of microscopic methods to the study of the structure of metals owes a good deal to the work of Osmond, and it was as a result of his interest in the study of steel by physical methods that he originated the allotropic theory of hardening, a theory which he supported up to the time of his death forty years ago.*

**T**HIS month sees the fortieth anniversary of the death of one of the greatest metallurgists of the 19th and 20th centuries, namely, Floris Osmond. In England, if not in France, the name of Osmond as a pioneer of metallographic methods in the examination of the structure of metals has been somewhat obscured by that of Sorby; the fact remains that the widespread application of these methods of investigation was, essentially, due to Osmond. He was a friend of Sorby, probably one of the first to recognise and proclaim the value of his work, which he did in the most unselfish way.

His birthplace was Paris, where he first saw the light of day in the year 1849. When he was a student at the École Centrale des Arts et Manufactures, his instructor was Professor Jordan, whose influence gave him that bias in the direction of metallurgy that remained with him all his life. On graduating, he obtained employment in the machine shops of the Fives Lille company, and thence transferred his services to the Compagnie Denain et Anzin. These large works were being reconstructed and undergoing a necessary expansion: included in the work was the introduction of equipment for the production of steel by the Bessemer and open-hearth processes. Osmond helped to start the open-hearth plant and, as a result, was able to collect practical experience in the manufacture of both iron and steel.

#### The Allotropic Theory of Hardening

In 1880, Osmond, still in pursuit of experience, joined the metallurgical staff of the great French armament and steel-making firm, Le Creusot. There he met Werth, with whom he collaborated in metallurgical research, and was thus the third scientist to take up the microscopic examination of steel. In 1885, he and Werth published a paper on "The Critical Points of Iron and Steel," and this formed the basis for the allotropic theory of hardening. Osmond came to the conclusion that "Hardened steel is a steel in which the iron and carbon have preserved more or less completely in the cold state the condition they possessed at high temperatures. The heat of the change that has not been effected remains disposable in the metal, and may be termed the latent heat of hardening. I conclude that the hardened steel owes its properties principally to the presence of beta iron, which is hard and brittle by itself at ordinary temperatures. Carbon, in a particular form maintains iron in the beta state during slow cooling down

to a temperature which is in inverse proportion to the amount of carbon contained in the steel. The influence of the carbon is of the same character as that of the rate of cooling, and both combine to produce the final result."

Osmond was, beyond question, the initiator of the allotropic theory, but he was not the only metallurgist operating along similar lines at that period. At an early date, in 1886, Professor H. Le Chatelier had introduced the thermo-electric pyrometer known to-day by his name, and in consequence had provided metallurgical research workers with a most valuable tool. In the same year, Osmond, taking advantage of this accurate method of measuring temperatures, set to work to study the deviations which had been noticed in the temperature curves of iron and steel on heating and cooling. It had already been suggested, over 20 years previously, by the Russian metallurgist Tschernoff and others, that these evolutions and absorptions of heat arose as the result of a change in the molecular pattern, and the cause of these phenomena had first been indicated by Barrett. It was, however, Osmond's brilliant interpretative faculty and careful research that revealed more clearly certain facts about these deviations, and led to the accurate investigation of the critical points or change points of iron and steel on heating and cooling, an investigation to which Osmond's paper: "The Phenomena which occur during the Heating and Cooling of Cast Steel" materially contributed. Later, he published a further contribution to the subject entitled "The Transformations of Iron and Carbon."

In the years directly succeeding these papers, the theory of the allotropic modification of iron and steel was largely accepted, and experimental work appeared to justify Osmond's claims. There were, however, metallurgists who took an opposing view, and as a result there came in time to be, in the international scientific community devoted to the study of iron and steel, a band of workers who believed in what was termed the "carbonist" theory. This, summarised, was the view that hardening arose as a result of the enforced solution of the carbon in iron by sudden cooling, the hardness being a direct function of the rate of cooling. Hardening was believed by these workers to be accompanied by great internal strains, and the molecular stresses produced hardness in a manner not unlike that of cold working. Between the two schools of thought there was unceasing argument—great metallurgical names parti-

cipating, e.g. Roberts-Austen, Hadfield, Arnold—with the result that one of the most famous scientific controversies of the time raged in every country interested in iron and steel. The Iron and Steel Institute's Alloys Research Committee tended to favour Osmond.

### The Application of the Microscope

Before dealing further with this dispute, however, we must return to Osmond's career. He was, primarily, interested in applying the microscope to the study of iron and steel, and in the preparation, polishing and etching of sections of metal for microscopic examination. He followed on Sorby and Martens by improving the methods of investigation, and during his investigations he was responsible for discovering a number of new constituents, namely, martensite and austenite, though at that time these constituents did not then possess their present names. He also recognised and gave their names to sorbite and troostite.

Even as late as 1912, the year of his death, Osmond continued to uphold his allotropic theory; but the controversial thicket is so tangled from 1890 onwards that to endeavour to penetrate it would confuse, embarrass and probably bore the reader. We will, therefore, confine ourselves to remarking that this battle of the theories had one bad effect. It made the practical men of the iron and steel industry doubt the value and use of metallography. On the other hand, it did a great deal to develop the still embryonic art of preparing and etching sections, brought about higher magnifications and improved photographic apparatus, and also induced metallurgists to approach the problem more scientifically. It should be pointed out that during this period (from 1890 to 1900) American voices began to be heard, Sauveur and Howe bringing material contributions to metallurgical research.

### Honours and Awards

In 1890, Osmond was elected a Member of the Iron and Steel Institute, and it was to this body in May of that year that he gave the paper on critical points referred to earlier. During his lifetime, he was not without ample recognition and reward for his brilliant work. Money prizes were given to him by the Société d'Encouragement pour l'Industrie Nationale in 1888 and 1895. In 1897 he was awarded the Lavoisier Medal, and in 1898, he was elected an Honorary Member of the American Institute of Mining Engineers. In 1906 he was given the Bessemer Gold Medal of the Iron and Steel Institute, but he was at that time far from well, and it proved impracticable for him to be present to receive the honour in person. It was, therefore, presented to the Ambassador of France, who forwarded it to Osmond.

During the same year he contributed his third and final paper to the Institute. This dealt with the "Crystrallography of Iron." He was an active correspondent, his pen being ever ready to defend the allotropic theory when attacked in the scientific and technical press. He died on June 18th, 1912, at his home at Saint Leu in the Department of Seine-et-Oise, France, at the age of 63, and was buried privately at the cemetery of Taverny, Seine-et-Oise, six days later.

This article would not be complete without reference to the book, published in 1904, entitled: "The Microscopic Analysis of Metals," in which Osmond collaborated with the late J. E. Stead. This work, which saw three

editions before being supplanted by later and more elaborate studies, contains two of his papers on Microscopic Assay, given to the International Association for the Testing of Materials in 1897 at the Stockholm Congress. It also contains a revised work on "The Micrographic Analysis of Carbon Steels," previously published by the Société d'Encouragement pour l'Industrie Nationale in 1895. The work concludes with a final chapter describing the micro- and photographic apparatus Osmond used, and the way in which he used it, together with a special note on austenite. Stead wrote of Osmond: "The careful and logical reasoning and the hypothetical conclusions arrived at have their charm, and no one can carefully read them without feeling that they have been made by a master mind, whose one aim is to arrive at the truth."

When the third edition was published in 1924, the Editors wrote in their preface: "It remains to add that, so far as the theory of the transformations of iron are concerned, Osmond, despite all assertions to the contrary, never altered his views from those announced in the original edition, and this fact appealed to the editors as an additional reason for leaving those portions intact. . . . In a letter written within a few weeks of his death . . . he" (Osmond) "expressed himself as follows: ' . . . my opinions have undergone no change whatever, despite the discussions which have taken place on the subject . . . chiefly I consider that nothing definite has been arrived at in regard to controversial points. . . .'"

It is interesting to remember that the name "metallography" was not given to the new science, in the founding of which Osmond played so large a part, until the year 1892. Osmond, as indicated, had contributed commemorative names to one or two new constituents of the structure of steel; but curiously enough, osmondite, a name which one would have thought applicable in his honour to some other constituent, never "caught on," when so applied by Heyn.

### Indian Photographic Tour

In what is believed to be a pioneer venture, Rooster Publicity Limited are arranging to send a fully-equipped photography unit to India and Pakistan for the purpose of obtaining first-class illustrations of British plant, machinery and capital goods, with a view to facilitating future export marketing. The unit will be leaving this country in the early part of September, 1952, and facilities will be available during the tour for taking a limited number of 16 and 35 mm. documentary films.

The unit is scheduled to travel over 40,000 miles and will be dealing with photographs of British equipment supplied to the major hydro-electric schemes, the river valley developments and many other industrial projects. By serving a number of British companies during the tour, it is hoped to bring the cost of photographs within a scale comparable to those adopted by the British Institute of Photographers for commercial photographs taken in Great Britain. Readers who may be interested in taking advantage of these facilities should get in touch with Rooster Publicity, Ltd., Walton House, Peterborough.

Further tours are being planned, covering Scandinavia, Spain and Portugal, the Middle East, Africa and other overseas territories which are of vital importance to the British exporter.

# The Annealing and Heat Treatment of Ductile Cast Iron

By C. C. Hodgson, A.Met., F.I.M. and W. Fairhurst, B.Sc., A.Met., A.I.M.  
*Leyland Motors, Limited*

*In this article are presented the results of an investigation undertaken to determine the relationship between the thermal treatments applied to ductile cast iron and its structure and mechanical properties. The treatments concerned comprise annealing, for the purpose of improving machinability or developing the maximum ductility and toughness, and hardening and tempering.*

In a previous article,\* Johnson and one of the present authors reported the results of some of their experiments made to establish the influence of variations in chemical composition, base material and other factors, on some of the properties of ductile cast iron. The authors now propose to give an account of the relationship between the thermal treatments applied to similar material and its structure and mechanical properties.

The experimental specimens were derived from quartered castings of the kind illustrated in Fig. 1, or smaller pieces machined from these. The castings were derived from 18-lb. high-frequency furnace melts, using as base a refined iron of the following analysis :

\* *Metallurgia*, May, 1952, pp. 218-224

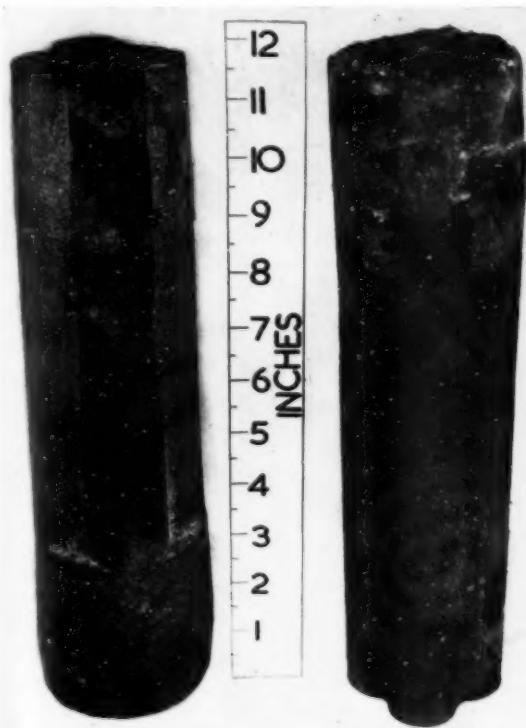


Fig. 1.—Test piece castings.

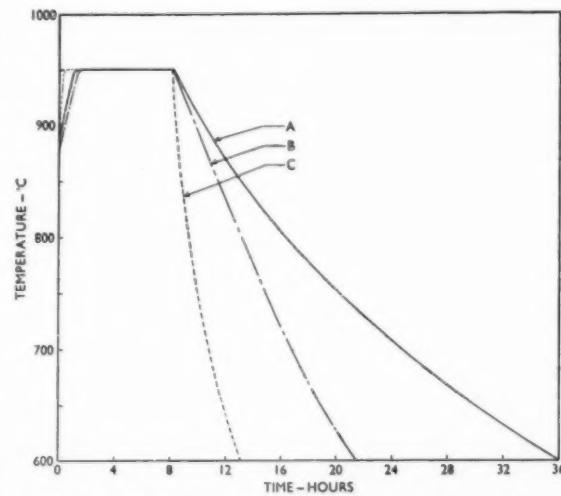


Fig. 2.—Heating and cooling cycles used for the determination of effect of rate of cooling.

Carbon	3.65%
Silicon	0.04%
Manganese	0.15%
Sulphur	0.01%
Phosphorus	0.03%
Nickel	0.04%
Chromium	0.02%
Copper	0.12%

Magnesium was added in the form of Mond Nickel No. 1 nickel-magnesium alloy. Most of the annealing was done under conditions that gave one of the cooling rates shown in Fig. 2.

## Annealing Ductile Cast Iron

Ductile cast iron may be annealed for one or both of two reasons: (1) to decompose any excess cementite that may be present in thin sections, to avoid machining troubles that would be caused by the presence of this constituent; and (2) to obtain the maximum ductility and toughness by developing an all-ferritic matrix.

It will be evident that if the sole object of annealing is to decompose cementite, the highest toughness and impact value and the attainment of highest possible machining speeds being relatively unimportant, then

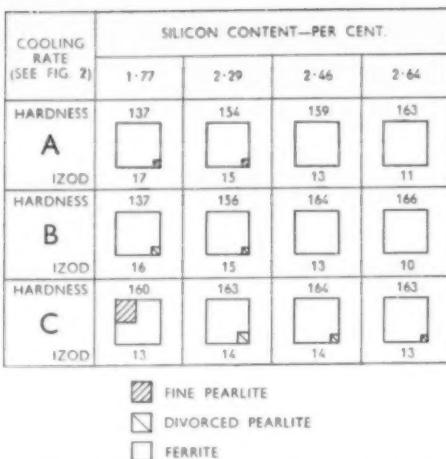


Fig. 3.—Effect of silicon content and cooling rate after annealing on the Izod impact and Brinell hardness (H.10/3000).

the rate of cooling after annealing is relatively unimportant also. If, on the other hand, the maximum attainable impact value is required, experimental evidence has shown it to be necessary to procure a ferritic matrix, as nearly as possible free from pearlite. Here the cooling rate is likely to exert an important influence. It is important in this connection to know the relationship between variations in silicon content and, more particularly, manganese content and cooling rate, and their effect on microstructure and, therefore, on impact value.

The following notes record the results of experiments made to establish the influence of cooling rate, silicon content, and manganese content on impact value and microstructure. All treatments used for the silicon series were of the conventional annealing type, i.e. heating, soaking and furnace cooling.

#### Relationship Between Silicon Content and Cooling Rate After Annealing, and Izod Impact and Hardness Number

Test bars of ductile iron from high-frequency casts made with silicon contents varying from 1.77-2.64% and otherwise to the preferred analysis,\* were annealed at 950°C. and cooled at standard rates A, B and C. The results obtained are illustrated in Fig. 3. The general influence of increasing silicon content is to assist the annealing process at the cost of increasing the hardness of the iron and reducing its impact value. The effect of variations in the cooling rate and silicon content on the microstructure of the iron can be seen in photomicrographs Figs. 4-7, all of which are of ductile iron of low manganese content annealed at 950°C.

#### Relationship Between Manganese Content and Cooling Rate After Annealing, and Izod Impact and Hardness Number

The influence of manganese has been investigated at somewhat greater length than that of silicon, and some of the more important results are given in Tables I, II and III. The results given in Table I suggest that the material is relatively insensitive to cooling rate over a wide range of manganese content. Whilst this is certainly true for low-manganese ductile cast irons, it is evident that when the manganese is high this insensi-

\* C, 2.9 to 3.2%; Mn, 0.3% max., P, 0.05% max.

TABLE I.—EFFECT OF VARIATION IN RATE OF COOLING AND MANGANESE CONTENT ON THE IMPACT VALUE AFTER ANNEALING FOR 8 HOURS AT 950°C.\*

Cast	HF120	HF71	HF70	HF99	HF140
Manganese %	0.08	0.25	0.44	0.68	0.82
Carbon %	3.00	2.81	2.98	3.00	3.12
Silicon %	1.98	1.92	1.96	1.96	2.00
Nickel %	0.94	0.90	0.96	0.94	0.91
Magnesium %	0.10	0.09	0.07	0.06	0.07
<i>Cooling Rate A (Fig. 2)</i>					
Izod Impact—ft. lb.	18, 17, 19	17, 16, 17	15, 16, 15	14, 13, 13	12, 12, 12
Average	18	17	15	13	12
Hardness H.10/3000	131	140	131-143	149-156	159
<i>Cooling Rate B (Fig. 2)</i>					
Izod Impact—ft. lb.	16, 16, 16	14, 15, 15	14, 14, 14	14, 15, 12	12, 12
Average	16	15	14	13	12
Hardness H.10/3000	134-137	140-143	143-146	159	156-159
<i>Cooling Rate C (Fig. 2)</i>					
Izod Impact—ft. lb.	15, 15	14, 15, 15	13, 13	11, 10, 11	10, 9
Average	15	15	13	11	10
Hardness H.10/3000	134	146	146-153	170-174	187

\* Mechanical tests on sections of cloverleaf test bars.

TABLE II.—EFFECT OF VARIATION IN MANGANESE CONTENT ON THE MECHANICAL PROPERTIES AFTER VARIOUS ANNEALING CYCLES\*

Cast	HF139	HF140
Manganese %	0.07	0.46
Carbon %	2.85	3.03
Silicon %	2.17	2.06
Nickel %	1.02	0.94
Magnesium %	0.09	0.10
<i>Annealed 8 hours 950°C. and Furnace Cooled at Rate A (Fig. 2)</i>		
Yield Stress—tons/sq. in.	20.3	20.4
Ultimate Tensile Stress—tons/sq. in.	27.2	28.6
Elongation %	22.5	22.6
Reduction of Area %	21.5	21.5
Izod Impact—ft. lb.	17, 17, 17	14, 14, 14
Average	17	14
Hardness H.10/3000	143-146	145-153
<i>Annealed 8 hours 950°C., Transferred to Furnace at 690°C., Held 4 hours and Furnace Cooled at Rate B (Fig. 2)</i>		
Izod Impact—ft. lb.	17, 16, 17	7, 8, 8
Average	17	8
Hardness H.10/3000	146	163
<i>Annealed 8 hours 950°C., Transferred to Furnace at 690°C., Held 4 hours and Cooled in Box in Air</i>		
Izod Impact—ft. lb.	17, 17, 17	7, 7, 7
Average	17	7
Hardness H.10/3000	149	179-183

\* Mechanical tests on sections of cloverleaf test bars annealed as indicated.

TABLE III.—EFFECT OF VARIATION IN COOLING RATE AFTER ANNEALING\*

Cast	HF149	HF124	HF150
Manganese %	0.06	0.31	0.82
Carbon %	2.86	2.99	2.82
Silicon %	2.07	1.98	2.07
Nickel %	1.02	0.88	0.98
Magnesium %	0.10	0.08	0.10
<i>Annealed 8 hours 950°C. and Furnace Cooled at Rate between A and B (Fig. 2)</i>			
Yield Stress—tons/sq. in.	19.4	20.2	22.0
Ultimate Tensile Stress—tons/sq. in.	27.2	27.7	31.9
Elongation %	24.0	18.0	17.5
Reduction of Area %	28.0	18.5	16.5
Izod Impact—ft. lb.	15, 16, 16	11, 15, 14	5, 6, 6
Average	16	13	6
Hardness H.10/3000	140-146	143	163-166
<i>Annealed 8 hours 950°C. and Furnace Cooled at Rate C (Fig. 2)</i>			
Izod Impact—ft. lb.	15, 15, 15	12, 12, 11	4, 4, 4
Average	15	12	4
Hardness H.10/3000	145-146	156-159	187-192

\* Mechanical tests on sections from clover leaf test bars heat treated as indicated.

tivity to cooling rate is likely to be upset by minor factors, as the results obtained from the two irons containing 0.82% manganese show (Tables I and III). These results emphasise the importance of keeping the manganese as low as possible if the material is intended for annealing.

The results in Table II were obtained when investigating the practicability of accelerating the annealing cycle by cooling quickly from the full annealing temperature to a temperature just below the  $Ar_1$  critical point. The low-manganese material is again shown to be insensitive to big differences in cooling rate, but the medium-manganese ductile cast iron must be cooled slowly after annealing if the best results are to be obtained.

Microscopic examination of the material of cast HF 140 (Table II), after the heat treatments referred to in the previous paragraph, established the fact that the object of cooling to a temperature just below the critical had not been attained. This may have been due to the rate of cooling of the hot box from the 950° C. furnace when placed in the furnace at 690° C., being so slow as it approached the latter temperature that its contents did not actually reach 690° C. during the 4 hours holding time, or to the  $Ar_1$  point being depressed by the 0.46% manganese present to a temperature slightly lower than the 690° C. used for heat treatment. Quite apart from such arguments an important fact is brought out: i.e. with manganese in the region of one-half of 1%, quick cooling after annealing can cause serious loss of impact value, even if the cooling is arrested in the region of the critical temperature. Photomicrographs of three irons of different manganese contents, after annealing at 950° C. and cooling at rate A (Fig. 2) are illustrated in Figs. 8, 9 and 10.

#### Changes During Cooling from the Annealing Temperature

In order to obtain information on the part played by cooling rate in achieving good impact resistance, a num-

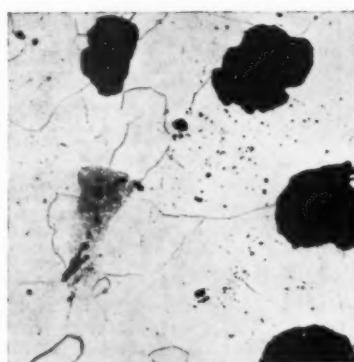
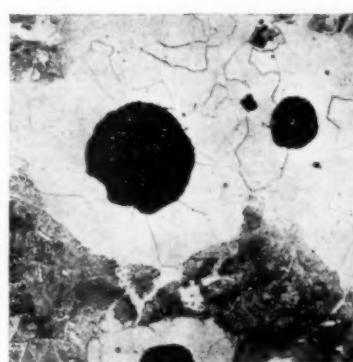
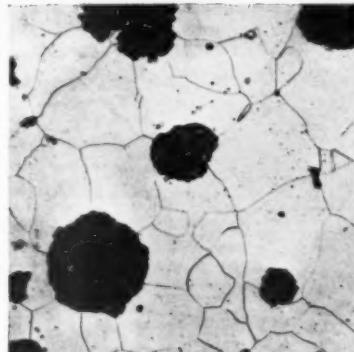


Fig. 4 (top left).—Si 1.77%. Cooling rate A.  $\times 200$ .  
 Fig. 5 (top right).—Si 2.64%. Cooling rate A.  $\times 200$ .  
 Fig. 6 (bottom left).—Si 1.77%. Cooling rate C.  $\times 200$ .  
 Fig. 7 (bottom right).—Si 2.64%. Cooling rate C.  $\times 200$ .

Figs. 4-7.—Effect of variation in silicon content and cooling rate on the microstructure of ductile cast iron of low manganese content annealed at 950° C.

ber of experiments have been made. In one series small specimens 0.45 in. diameter were annealed at 950° C. and cooled at rate A (Fig. 2), specimens being withdrawn and water quenched at various temperatures between 800° and 650° C. In a second series, similar specimens were normalised from 950° C. and then submitted to standard annealing, sub-critical annealing at 675° C., or

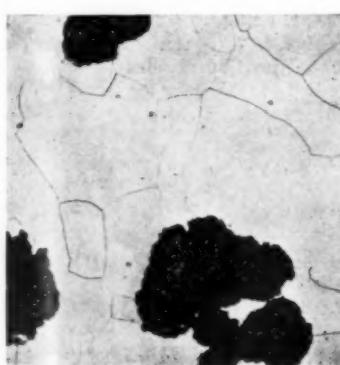


Fig. 8.—Mn 0.10%. Cooling rate A.  $\times 200$ .

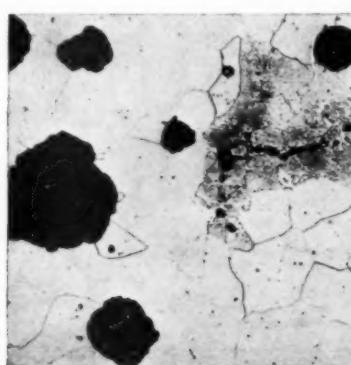


Fig. 9.—Mn 0.44%. Cooling rate A.  $\times 200$ .

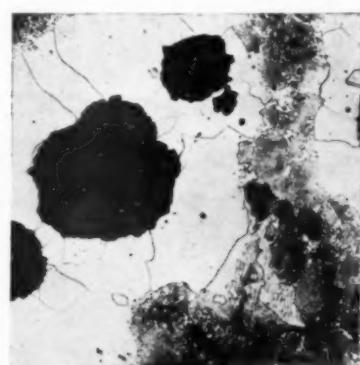


Fig. 10.—Mn 0.82%. Cooling rate A.  $\times 200$ .

Figs. 8-10.—Effect of variation in manganese content on the microstructure of ductile cast-iron annealed at 950° C. and cooled at rate A.

QUENCHING TEMPERATURE - C.	SILICON CONTENT—PER CENT.				
	1.5	2.0	2.3	3.0	3.2
800		■		■	
750	■	■	■	□	□
725	■	□	□	□	□
700	□	□	□	□	□
675	□	□	□	□	□
650		□			

■ MARTENSITE    ▨ PEARLITE    □ FERRITE

Fig. 11.—Effect of variation in silicon content on the changes occurring during slow cooling from 950° C. Sections cooled from 950° C. at rate A and water quenched from the temperature indicated.

isothermal treatment by quenching in a bath of molten aluminium at 675° C. from an initial temperature of 950° C.

The results obtained from the first series are shown in Figs. 11 and 12 for ductile cast iron of varying silicon and manganese content respectively. The temperature of the austenite  $\rightarrow$  pearlite change is raised by the

TABLE IV.—CHANGES OCCURRING DURING COOLING FROM THE ANNEALING TEMPERATURE\*

Treatment	0.13% Manganese	0.45% Manganese
(1) Normalised 950° C.	Martensite and troostite.	Martensite.
(2) Normalised 950° C. (slower cooling rate than (1)).	Matrix of pearlite with about 10% ferrite in bulls-eye form round the graphite nodules (Fig. 13).	Matrix of pearlite with a little ferrite.
(3) Fully annealed, slowly cooled to 675° C., then air cooled.	Matrix of ferrite with slight traces of pearlite.	Matrix of ferrite with a few areas of laminated and divorced pearlite.
(4) Quenched from 950° C. into molten metal bath at 675° C., held for 5 hours at 675° C. and air cooled.	Matrix entirely fine divorced pearlite (Fig. 14).	Matrix entirely divorced pearlite.
(5) Normalised 950° C. as for (1), reheated for 5 hours in molten metal bath at 675° C. and air cooled.	Matrix of ferrite with traces of pearlite present. A large number of very fine graphite particles in addition to the usual graphite nodules (Fig. 15).	Matrix of ferrite with 15–20% pearlite present. A large number of very fine graphite particles in addition to the usual graphite nodules.
Normalised 950° C. as for (2), reheated 5 hours in molten metal bath at 675° C. and air cooled.	Matrix of ferrite with 35–40% pearlite present (Fig. 16).	Matrix of ferrite with 20–25% pearlite present.

Nominal Composition: 3% Carbon, 2% Silicon.

\* All specimens 0.45 in. diameter given a preliminary full anneal at 950° C. prior to machining.

QUENCHING TEMPERATURE - C.	MANGANESE CONTENT—PER CENT.				
	0.13	0.28	0.44	0.68	0.82
800	■	■			
750	■	■	■	■	■
725	□	□	■	■	■
700	□	□	□	■	■
675	□	□	□	□	□
650	□	□	□	□	□

Fig. 12.—Effect of variation in manganese content on the changes occurring during slow cooling from 950° C. Sections cooled from 950° C. at rate A and water quenched from the temperature indicated.

addition of silicon and lowered by the addition of manganese.

The second series of experiments produced some interesting results which have been summarised in Table IV: photomicrographs illustrating some of the structures are shown in Figs. 13–16. Their full elucidation would need considerably more work.

Free air-cooling of the small specimens from 950° C. produced a matrix of martensite, or of martensite and troostite. Subsequent sub-critical annealing of such specimens led to the formation of a large number of small graphite particles not previously observed (Fig. 15). When the specimens were cooled at a slightly slower rate from 950° C. the matrix consisted mainly of laminated pearlite (Fig. 13). Sub-critical annealing of these specimens produced no fine graphite, the matrix was now made up of ferrite and partly divorced pearlite (Fig. 16). Thus, differences of constitution before applying sub-critical annealing lead to differences in structure after this treatment.

The way in which the small specimens were caused to approach the sub-critical annealing temperature also influenced the results obtained. When material in the austenitic condition was brought quickly to the sub-critical annealing temperature, and held there, divorced pearlite was formed (Fig. 14) but, as previously mentioned, ferrite and fine graphite were produced when martensitic material was raised quickly to the sub-critical annealing temperature (Fig. 15).

#### Sub-Critical Annealing

Ductile cast iron after sub-critical annealing is usually less tough and slightly harder than after conventional

annealing. The results of Izod and hardness tests on three irons of different manganese contents are given in Table V, together with the corresponding results after conventional annealing for comparison.

### Influence of Minor Elements

Experiments were made on three casts of magnesium-treated iron containing small amounts of lead, antimony and tin to determine the effect of cooling rate after annealing. Tin makes the ductile cast iron susceptible to cooling rate after annealing. Lead and antimony ruin it. Some Izod impact values obtained from these irons are given in Table VI.

### Hardened and Tempered Ductile Cast Iron

Hardening and tempering experiments were made on specimens machined from clover leaf test bars derived from laboratory melts. The specimens were approximately  $1\frac{1}{2}$  in. in diameter at the stage of oil quenching. The results obtained will be found in Table VII, and the mechanical properties of the corresponding fully annealed irons are included for comparison. Photomicrographs of the irons in the hardened and tempered condition are illustrated by Figs. 17-20.

An examination of the mechanical test results shows that in the annealed condition the iron with the higher silicon content has also the higher tensile strength; this would be expected from the analyses of the two irons. After hardening and tempering the reverse holds true, the reason for this being made clear by their micro-structures. Dispersed throughout the matrix of the

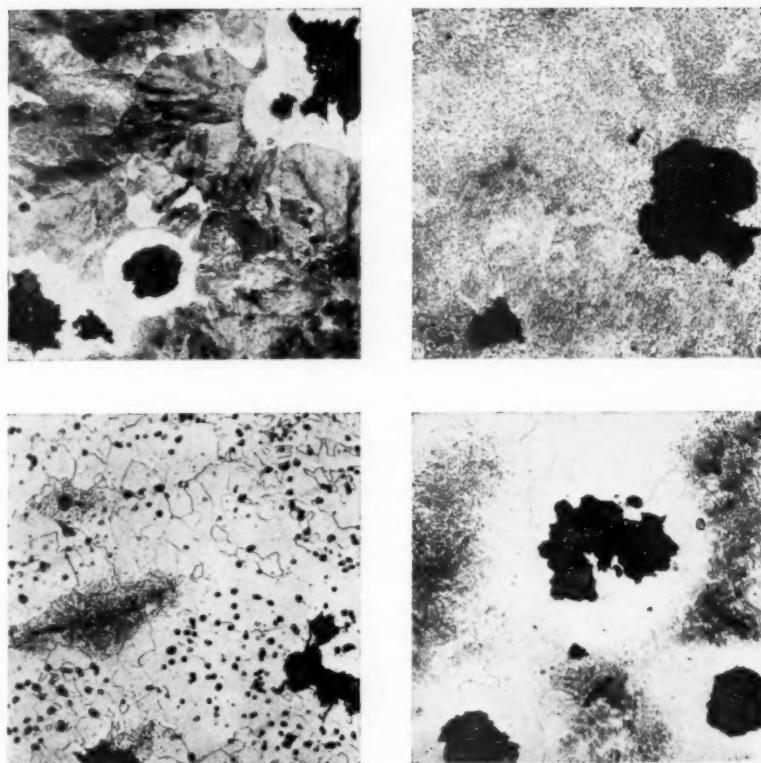


Fig. 13 (top left).—Normalised 950° C.—slightly slower than free air cooling. Matrix of pearlite with about 10% of ferrite in bulls eye form round the graphite nodules.  $\times 200$

Fig. 14 (top right).—Quenched from 950° C. into a molten metal bath at 675° C., held for 5 hours and air cooled. Matrix entirely fine divorced pearlite.  $\times 200$

Fig. 15 (bottom left).—Normalised 950° C.—free air cooling—reheated for 5 hours in a molten metal bath at 675° C., and air cooled. Matrix of ferrite with traces of pearlite present. Large number of very fine graphite particles in addition to the usual nodules.  $\times 200$

Fig. 16 (bottom right).—Normalised 950° C.—slightly slower than free air cooling—reheated for 5 hours in a molten metal bath at 675° C., and air cooled. Matrix of ferrite with 35-40% pearlite present.  $\times 200$

Figs. 13-16.—Effect of normalising, followed by sub-critical annealing and isothermal treatment on the microstructure of ductile cast iron containing 0.13% Mn and nominal 3% C and 2% Si.

TABLE V.—COMPARISON OF IMPACT AND HARDNESS FIGURES AFTER NORMAL AND SUB-CRITICAL ANNEALING

Treatment	Property	Manganese 0.06%	Manganese 0.31%	Manganese 0.82%
Annealed 8 hours at 950° C. and furnace cooled at Rate A (Fig. 2)	Izod Impact—ft. lb. Average	16, 16, 16 16	11, 15, 14 13	5, 6, 6 6
	Hardness H/10/3000	140-146	113	163-166
Sub-critical annealing 12 hours at 710° C. and furnace cooled at Rate A (Fig. 2)	Izod Impact—ft. lb. Average	12, 13, 12 12	9, 8, 7 8	3, 3, 3 3
	Hardness H/10/3000	153-156	156-159	159-163

TABLE VI.—IZOD IMPACT TEST RESULTS (FT. LB.) ON DUCTILE CAST IRON CONTAINING LEAD, ANTIMONY AND TIN

Cooling Rate (Fig. 2)	Lead 0.02%	Antimony 0.08%	Tin 0.09%
Rate A ...	3, 3, 3	3, 4, 4	13, 13, 12
Faster than Rate A but slower than Rate B ...	3, 3, 3	4, 3, 5	6, 6, 5

TABLE VII.—MECHANICAL PROPERTIES OF HARDENED AND TEMPERED IRONS\*

Cast	HF80		HF89	
	Annealed 8 hours at 950° C. and furnace cooled at Rate A (Fig. 2)	Annealed, Oil Quenched 900° C. and Furnace Cooled at Rate A (Fig. 2)	Annealed 8 hours at 950° C. and furnace cooled at Rate A (Fig. 2)	Annealed, Oil Quenched 900° C. and furnace cooled at Rate A (Fig. 2)
Carbon %	3.25	3.06	3.06	2.06
Silicon %	1.48	0.11	0.13	—
Manganese %	0.11	0.010	0.020	0.030
Sulphur %	0.010	0.026	0.026	0.020
Phosphorus %	0.010	0.018	0.018	0.018
Nickel %	0.78	0.78	0.96	0.96
Magnesium %	0.10	0.10	0.09	0.09
Heat Treatment				
Yield Stress—tons/sq. in.	15.8	26.0	19.4	34.0
Ultimate Tensile Stress—tons/sq. in.	24.2	49.0	26.6	42.6
Elongation %	25.0	7.0	26.0	7.5
Reduction of Area %	24.0	5.0	32.0	6.0
Izod Impact ft. lb. Average	18, 18, 18	7, 7, 6	15, 16, 16	5, 6, 5
Hardness H/10/3000	131	235	149	190-202

\* Mechanical tests on sections of clover leaf test bars heat treated as indicated.

higher silicon iron, HF 89, are a large number of small particles of graphite. It would appear that these have been formed during the tempering operation at the expense of the carbon content of the sorbite derived from the tempered martensite. The carbide depletion can be seen if Figs. 18 and 20 are compared, the latter also illustrates the fine particles of graphite in the hardened and tempered matrix.

It was desired to produce a few experimental parts from hardened and tempered castings. The results of tests obtained from ductile cast iron produced from all-steel charges, or from steel and ductile cast iron scrap, melted in the basic cupola are given in Table VIII. The percentage of silicon present, it will be noticed, is rather low. This was intentional in view of the knowledge of the influence of this element on changes taking place during tempering, and so on tensile strength, obtained from the preliminary experiments.

#### Hardenability Tests

Jominy end-quench hardenability tests have been made on three specimens of ductile cast iron melted in a basic lined cupola. The test pieces were machined from clover leaf test blocks which had been previously annealed by heating for 8 hours at 950° C. followed by slow cooling (cooling rate A, Fig. 2). The machined specimens were then packed in cast iron borings, re-heated to 900° C., soaked at this temperature for 20 minutes and end quenched by water. Two diametrically opposite flat surfaces were ground and polished and diamond pyramid hardness tests carried out in the normal manner for tests of this type.

The analyses and the more important points of the hardenability curve are given in Table IX.

The examination of sections cut from hardened and tempered castings gave the impression that the practical effect of oil-quenching ductile cast iron is a less intense

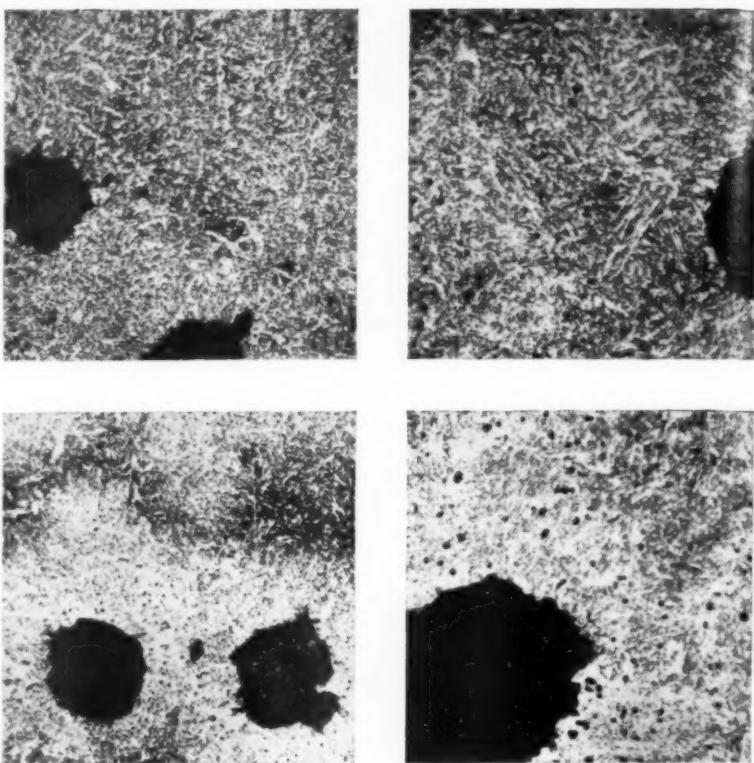


Fig. 17 (top left).—Si 1.48%.  
Fig. 18 (top right).—Si 1.48%.  
Fig. 19 (bottom left).—Si 2.06%.  
Fig. 20 (bottom right).—Si 2.06%.

Figs. 17-20.—Effect of hardening and tempering on the structure of ductile cast iron. Specimens annealed 950° C., oil quenched 900° C., and tempered 600° C.

hardenability than a consideration of the Jominy figures might lead one to expect. Transformation products made their appearance at comparatively small distances below the surface in hollow castings of about 1½-in. wall thickness, casting weight about 76 lb.

#### Summary and Conclusions

If the object of annealing ductile cast iron is merely to decompose the pearlite (and any cementite that may be present) then it is apparent that a high silicon content

TABLE VIII.—MECHANICAL TESTS ON HARDENED AND TEMPERED DUCTILE CAST IRON<sup>a</sup>

Identification Mark	D.155	D.160	D.162
Carbon %	3.63	3.1	3.21
Silicon %	1.72	1.44	1.32
Manganese %	0.25	0.22	0.22
Phosphorus %	0.022	0.036	0.032
Nickel %	1.58	Not determined	Not determined
Chromium %	0.06	trace	trace
Magnesium %	0.09	0.09	0.11
Heat Treatment	O.Q.900° C. T.630° C.	O.Q.900° C. T.600° C.	O.Q.900° C. T.900° C.
Yield Stress—tons/sq. in.	36.8	36.8	40.2
Ultimate Tensile Stress—tons/sq. in.	46.0	45.0	47.8
Elongation %	0.5	—	—
Izod Impact—ft. lb.	4, 4, 4	4, 4, 4	3, 3, 3

<sup>a</sup> Section heat treated—approximately 1½ in. diameter cut from clover leaf test block previously annealed from 950° C. Standard 0.564 in. diameter tensile test piece.

TABLE IX.—DETAILS OF HARDENABILITY TESTS.

Specimen	D150	D151	D155
Carbon %	3.00	3.52	3.63
Silicon %	1.29	1.69	1.72
Manganese %	0.27	0.30	0.25
Sulphur %	0.020	0.016	0.020
Phosphorus %	0.032	0.036	0.022
Nickel %	1.76	1.08	1.58
Chromium %	0.04	0.05	0.06
Magnesium %	0.06	0.10	0.09
Hardness HD/30			
(a) Maximum	621	655	606
(b) At 2½ in. from water-quenched end	283	298	274
(c) At point of inflection	445	418	418
Distance of point of inflection from water-quenched end— inches	0.75	0.96	0.90

Continued on page 296

# Steelmaking Topics Discussed

## Report of B.I.S.R.A. Conference

*At the 38th Steelmaking Conference, organised by the Steelmaking Division of the British Iron and Steel Research Association, and held at Ashorne Hill recently, the topics for discussion included the effect of increased pig iron to scrap ratio on steelmaking practice; the reduction of the metallurgical load on the open hearth furnace; experiments on feeder heads; basic refractories in ladles; and the effect of teeming speed on ingot quality. A brief account of the proceedings of the conference is presented here.*

THE maintenance of steel output and quality, in spite of necessary increases in the proportion of pig iron used in steelmaking furnaces because of lower scrap supplies, was one of the themes of the 38th Steelmaking Conference, held at Ashorne Hill, Leamington Spa, on May 7th and 8th last. The conference was, as usual, organised by the Steelmaking Division of the British Iron and Steel Research Association. Dr. Colclough, C.B.E. (British Iron and Steel Federation), was in the Chair and Mr. R. Lamb (Hadfields Ltd.) acted as Vice-Chairman.

### The Effect of the Ratio of Pig Iron to Scrap on Hot Metal Practice

In the first paper, Mr. J. S. Curphey, of Dorman Long and Co. Ltd., spoke on "The Effects of Increasing the Proportion of Iron used in the Fixed Open Hearth Practice at Redcar Melting Shop."

At Redcar, the percentage of iron to the furnaces was increased from 54% to 72% during the course of 1951, the usual proportions before that having been about 50%. Because the Redcar blast furnace could not provide all the extra iron, a vigorous recovery drive at the slag and ballast tips was necessary to obtain iron and steel skull, which was charged through the mixer in addition to a quantity of cold plate iron which was available within the company at that time.

The 350-ton active mixer is oil fired and its limited capacity gave rise to some difficulty as the weekly throughput rose to 7,500 tons, including 1,850 tons of cold metal. Though the average analyses indicated the removal of 27% of the silicon, 20% of the sulphur and 10% of the phosphorus from the molten iron from the blast furnaces, unpredictable fluctuations in the analysis of the iron to the furnaces led, at times, to hard or soft melts and melting slags of variable basicity.

*Slag Bulk.*—In discussing problems raised by the greater slag bulk resulting from higher iron/scrap ratios, Mr. Curphey said that if the common assumption that the density remains constant were allowed, the change from 54% to 72% of iron in the furnace charges at Redcar would mean an increase of 40% in the slag volume, equivalent to an extra 1½ in. in the slag depth. Assuming a temperature gradient through the slag of 10° C./inch this would mean an increase of about 18° C. in the slag surface temperature for a given metal temperature. He thought, however, that such assumptions were unwarranted, since higher percentages of hot metal often promoted a tendency towards increased porosity of the slag at a period in the charge when the rapid transfer of heat to the bath was essential and, although the effect of slag weight on the heat transfer

rate was considerable, the effect of density was of even greater magnitude. Under certain circumstances a given weight of slag might be subject to a considerable increase in porosity together with a corresponding increase in depth, and few steelmakers were fortunate enough to be unaware of the insulating properties of a foaming slag. Such slags might be a major problem in shops using uncarburetted mixed gases, or other fuels producing flames of low luminosity, but serious foaming should be the exception rather than the rule in oil-fired furnaces, and such had been the case at Redcar with the hot metal percentages so far experienced.

*Flush Practice.*—To minimise the disadvantages of increasing slag bulk, a more regular flush practice was adopted. Studies of slag compositions at Redcar had shown that the optimum time for slagging was between one and two hours after the furnace was full. The iron and manganese contents of the flush slag at that period did not represent a total loss, since the removal of appreciable quantities of  $P_2O_5$  and  $SiO_2$  resulted in a reduced weight of refining slag and, consequently, a lower weight of iron and manganese in the slag run off after tapping.

*Roof Wear.*—To offset the effect of slag surface temperatures on the upper furnace refractories, some of the silica in the front lining of the furnaces had been replaced by basic bricks, and trials with higher quality silica roof blocks, mainly in the area of maximum wear adjacent to the back skew, were in progress and results were encouraging.

The skewback castings on six furnaces with fully sloping back walls were lifted 9 in. to bring them in line with the front. These and other modifications connected with burner design and positioning had to be borne in mind when considering the effects of higher hot metal percentages on refractories wear, and with only two roofs annually per furnace, long periods were necessary for accurate assessment of the effects of modifications in practice. Results to date, however, indicated that roof lives were being steadily improved.

*Fuel Consumption.*—There had been little change in fuel consumption in spite of altered practice. Extra hot metal had offset the heat transfer difficulties, longer refining times and the additional oxides and fluxes. The shop fuel consumption, moreover, was affected by the fact that the additional metal was mainly made up of cold plate iron. Both shop and furnace oil consumptions were maintained at a fairly steady figure of 26 gallons/ton for the furnaces and 29 gallons/ton including the mixer: these figures included lighting-up fuel.

During the previous year steps to improve combustion practice had met with some success and the continuation



Fig. 1.—High silicon blocks near back skew on furnace at Redcar Works.

of these efforts during 1951 might have had some influence on the results obtained.

**Fettling.**—Heavier fettling, due to the higher acidity of the early slags followed by longer refining times at high temperature, was expected. Steps were, therefore, taken to maintain the grading of the crushed Doloma closer to the optimum, and though bad bottoms and banks became more frequent, the average fettling times and Doloma consumption did not show any appreciable increase.

**Production.**—Though the annual level of production was maintained, weekly fluctuations were somewhat more marked than they had been with 50/50 practice, partly because furnace performance was more sensitive to changes in iron quality. Developments in plant and practice had made it reasonable to expect that the progressive increase in production during the previous year would have continued, had sufficient scrap been available.

Since the period under review, shortage of materials had become even more acute, sometimes necessitating a reduction in the number of furnaces operating, and with furnaces standing empty for long periods waiting for charging materials production had fallen.

**The Future.**—Concluding, Mr. Curphey said that if the loss of scrap were replaced by an adequate supply of iron, there were various means of nullifying the disadvantages of the higher iron/scrap ratio and taking full advantage of the shorter charging times and greater sensible heat input. Among the alternatives under consideration were :—

- (1) Expansion of tilting furnace practice :
- (2) Controlled refinement of molten iron by :
  - (a) provision of adequate mixer capacity ;
  - (b) oxygen blowing of the iron in the ladle or mixer ;
  - (c) air blowing with oxide treatment in the ladle or mixer ;
  - (d) agitation of the metal in a reaction vessel together with the appropriate chemical treatment ;
  - (e) soda ash treatment ;

- (f) Bessemer/open hearth duplex in, using in the open hearth :
  - (i) fully blown metal plus mixer or blast furnace iron ;
  - (ii) partially blown metal plus scrap ;
  - (iii) fully blown metal (rebarred) plus scrap.

In any system of iron refinement, adequate facilities must be provided for the efficient removal of the slags formed before charging the metal to the furnaces. Though some of the above processes offered possibilities of considerable increases in the production rate, the choice must be largely influenced by economic factors and the availability of materials.

Steelmakers throughout the country might lament the passing of the scrap age, but greater difficulties had been overcome in the past and there was little doubt that, given an adequate supply of iron and facilities for its controlled refinement before charging to the furnaces, record breaking production rates could be achieved in the hot metal shops of this country.

#### The Effect of the Ratio of Pig Iron to Scrap on Cold Metal Practice

Mr. A. I. Aitken of Colvilles Ltd. presented a paper on the effect of an increased proportion of pig iron on cold metal practice. He took as examples three works with cold fixed furnaces, during three periods in which the first was "normal," previous to the reduction in scrap supplies, with a normal analysis pig range (phosphorus about 1.0%–1.3%); in the second and third periods the pig/scrap ratio had increased, but in the second the quality of the pig iron had deteriorated (phosphorus about 1.9%) and in the third it had recovered to 1.2%–1.4%.

**Liquid Fuel Fired Furnaces.**—Shop X contained 80-ton liquid fuel fired furnaces (nominal capacity around 65 tons) making about 35% of alloy, rimmed and free-cutting steels. The scrap lately used for alloy charges was nickel-chrome scrap, which "always adds another hour to melting time." In this shop, process time had increased by 20% and 13% from the first to the second and third periods respectively. This was equivalent to a decrease in productivity of 18% and 12%. Fuel consumption had risen inordinately and was in direct relation to exceptional pitside delays arising from plant alterations and the consequent reflection on the higher fettling times which were fuel consuming but nevertheless non-productive. There was also the greater reduction in heat transfer rates with liquid fuel and heavier slag layers in comparison with producer gas. Lime and dolomite consumptions had risen in accordance with the increased metalloids but the dolomite consumption was also a reflection of the longer operating time at high temperature.

**Gas Fired Furnaces.**—Shop Y contained producer gas fired 90-ton and 100-ton furnaces, the larger being fast-working Venturi furnaces. The results were similar to Plant X but, on the whole, to a less marked degree. Dolomite and lime followed the same pattern but the effect of reduced charging time was much more marked,

this offsetting the increased melting and refining times. The decrease in productivity was therefore less evident in the larger capacity furnaces. With the better quality pig burden there was barely any decrease in output, and only a slight rise in fuel over that obtaining during the optimum period.

Plant Z contained 90-ton and 150-ton furnaces, all fast-working Venturi types. The 90-tonners definitely showed a falling off in productivity with concomitant time, lime, dolomite and fuel increase, but the 150-tonners, probably from the direct influence of decreased charging time, showed a nominal decrease in productivity during the worst period and an actual increase during the third period.

Although it had been impossible to segregate fuel consumption for the different sizes of furnace in the producer gas fired shops, because of producers and mains layout, the speaker thought that the larger furnaces might actually show a lower fuel consumption per ton than during the optimum period.

It was clear that the charging of a furnace had a very great influence on its performance in altered conditions.

*Increased Yield.*—Commenting on the large increase in reducing elements used, Mr. Aitken said that this was additional tonnage at very little cost. Yields at all plants had been enhanced up to 5%. It might be possible, he thought, to charge much more ore and with the addition of suitable carbon, obtain direct reduction in the open hearth with increased yields and little, if any, increase in process times. Low ash carbon, such as anthracite fines briquetted with a suitable ore or scale, could, for a specific size of charge, replace part of the pig with consequent reduction on actual charge cost and gratuitous increase in yield.

*Metalloid Removal.*—Mr. Aitken thought that the biggest bugbear in fixed open hearth furnaces was phosphorus. "It is virtually impossible nowadays," he said, "to catch the higher carbons on the drop when one has a phosphorus specification to meet." Some pre-open hearth method of phosphorus removal was needed, and this was a subject that B.I.S.R.A. might usefully pursue. Sulphur raised few problems under present circumstances because of more dilution by large slag bulks.

He also thought that the new circumstances, which held molten metal and high oxide slags in contact with working hearths at high temperatures, made desirable better bottom making materials than rammed tarred dolomite hearths. Graphitised dolomite bottoms or a "fritted in" magnesite bottom might help to this end.

*Fuel Consumption.*—It was clear that, in fuel consumption, liquid fuel shops had been more adversely affected than gas fired shops. This might be because the transfer of heat from flame to stock with liquid firing was just as much by convection as it was by radiation, whereas with producer gas, heat transfer was much more by the radiation from the protracted combustion of a highly emissive flame. Large slag bulks would reduce this convective factor much more than the radiative factor. Nevertheless, because of the intrinsically lower flame temperatures developed, lean gas fired furnaces would always have a lower production potential than liquid fuel furnaces.

*Furnace Design.*—In considering furnace design Mr. Aitken said that as heat transfer occurred mainly by

convection and to a less extent by conduction, the heat transfer rate would be only slightly impaired by increased slag thickness. The deeper bath in the 150-ton furnace at plant Z (which had shown an increased output and only a small increase in fuel consumption) had meant that the increased pig/scrap ratio had had little effect on heat transfer rates. One thing at least was clear: the time formerly spent in charging was unnecessarily long, and ideas on the heat acceptivity rate of the material charged must be reviewed.

#### Joint Discussion

In the course of the discussion that followed these two papers it appeared that the increased pig iron/scrap ratio shortened charging and melting times and increased refining time. This meant that in hot metal shops there was little change in output, there had even been some increases; but in cold metal shops where refining time had previously been very short indeed, outputs were now reduced.

The increase in slag bulk raised many problems, and some interesting solutions were put forward. One works using direct blast furnace metal with wide silicon fluctuation "decanted" the blast furnace metal into a second ladle containing slag formers including fused soda blocks. When the slag had formed (in seven to ten minutes) oxygen was injected for a further ten minutes and 25% of cold pit scrap was added. The temperature had increased from 1,340° C. to 1,530° C. and a consistent silicon and sulphur content was secured, the latter having been reduced by half. This was now regular practice and had reduced slag volume considerably with a resultant gain in output.

One speaker described a convenient flushing technique developed in a hot metal melting shop originally designed for 70% scrap charges, but now using 30% scrap. Here 10 to 15 tons of slag was flushed off during each charge and poured into water tanks below the stage where it granulated and was subsequently fed by chutes to wagons.

In summing up the discussion the Chairman said that furnace design was ahead of shop design and some of the difficulties, such as that of greater slag bulk, could best be tackled by concentrating on shop design.

He asked those present what they would do when even less scrap was available and the pig iron/scrap ratio had to go even higher. The projected increase in steel output from 16 million tons to 20 million tons would mostly have to come from pig iron. It appeared that the staple ratio would be about 56% of iron to 49% of scrap overall and it was the task of those in authority to establish a reasonable balance in capacity and distribution of available materials. For that reason more tilting furnaces were now being constructed.

#### Reducing the Metallurgical Load

Mr. C. E. A. Shanahan (British Iron and Steel Research Association) described some of the work carried out for the Open Hearth Raw Materials Group of the Steel Practice Committee on ways of reducing metallurgical load.

Apart from slag flushing, the only practical means of avoiding an increase in open hearth slag bulk, and at the same time coping with a general increase in metallurgical load was, either to remove some of the metalloids from the blast furnace metal prior to the

open hearth furnace, or to continue refining the steel after the open hearth treatment. The latter methods, such as the Perrin technique, required that the metal should be poured through a separately melted slag from a considerable height. This had obvious disadvantages with large metal tonnages.

*Silicon, Manganese, Phosphorus.*—In concentrating on pre-open hearth refining treatments, study had been made of mixer data from several works. The general variability of the data had made it difficult to draw conclusions, but it had been found that there was a connection not only between the oxygen equivalent of silicon, manganese and phosphorus removed and the weight of lime added during the same period, but also between the oxygen equivalent of silicon, manganese and phosphorus removed and the number of disturbances occurring within the mixer during the same period. This suggested that in some mixers increased amounts of manganese, phosphorus and silicon could be removed in a given time by increasing the amount of agitation of slag and metal. Some preliminary experiments had been undertaken by one firm using air lances and although a slight increase in metalloid removal occurred the results were inconclusive because of the occurrence of other changes in conditions beyond control.

*Sulphur.*—The removal of sulphur in the open hearth furnace, while often adequate for high carbon steel production, could be a very serious problem for low carbon steel manufacture, especially in the production of deep drawing steel. As far as post-open hearth treatment was concerned, Canadian workers had recently performed some small scale trials on desulphurising molten steel with magnesium aluminium alloys and this process might be applied to large tonnages of steel in the teeming ladle under conditions where very low final oxygen contents were permissible.

The pre-open hearth desulphurisation of blast furnace metal had much to commend it, because the high carbon content of the metal was conducive to high sulphur partitions and therefore to slag and heat conservation; it also resulted in the sulphur becoming more chemically active and more easily removed by basic slags. Thirdly, the relatively low temperature of blast furnace metal and the high manganese content could, under certain conditions, result in the loss of sulphur from the metal by the mechanism of manganese sulphide flotation.

Statistical examination had been made of data obtained from seven mixers (four active and three inactive). In all cases but two, the amount of desulphurisation was small. In one of these two, it appeared that desulphurisation occurred rather by the separation of manganese sulphide than by the exchange of sulphur atoms in the metal with oxygen atoms in the slag, although the situation was complicated by the presence of  $\text{Na}_2\text{O}$  in the slag (from pre-mixer soda-ash treatment). The latter mechanism was considered to predominate in the remaining mixers, a reaction which was favoured by high slag basicity and low iron oxide content. The second mixer in which desulphurisation occurred was a good illustration; in this case desulphurisation efficiency was produced by the feeding of lime only. There was a tendency for sulphur to be removed from the bath by the waste gases, and although a particular slag might have a very low sulphur partition, important losses of sulphur might occur to the atmosphere.

The loss of sulphur by manganese sulphide separation could probably be used to greater effect in practice.

This would involve increasing the manganese content of the blast furnace metal in some way, e.g., by the addition of ferro-manganese or manganese oxide, and operating the mixer at as low a temperature as possible. To desulphurise, to any extent, without the manganese reaction required a high slag basicity and a very low iron oxide content, but these conditions were of no value for desiliconisation, which is essentially an oxidation reaction.

Another possible practical proposition was that a means of further desulphurising blast furnace metal would be to prolong the time of contact between metal and slag and at the same time to avoid large temperature drops. The fact that blast furnace slags were not in equilibrium with the metal had been shown in work by Hatch and Chipman, and by Darken and Filer.

Work by B.I.S.R.A. at Sheffield had been directed to the desulphurisation of carbon-saturated iron by blast furnace type slags. Preliminary studies had been mainly concerned with the effect of mixing on the rate of desulphurisation.

*Experimental Furnaces.*—It could be generally concluded that blast furnace or similar slags should be capable of rapidly desulphurising blast furnace metal, provided that highly reducing conditions and temperature could be maintained and that some means was available for intimately mixing the slag and metal together. A carbon-lined cylindrical furnace heated by a carbon rod resistor had been built (capacity 10 lb. of metal) which has a carbon wedge running longitudinally along the bottom of the melting chamber. The furnace could be rocked so that slag and metal continually cascaded over the wedge. It was intended to use this furnace for desulphurising carbon-saturated iron with blast furnace slags, and with lime-alumina non-siliceous slags. It was also intended to try other types of stirring furnace, such as a cylindrical vessel with a number of wedges or other obstructions which could be oscillated or rotated.

Going a stage further, blast furnace type slags had been desulphurised by blowing with air through a silica lance to see if their efficiency in removing sulphur from the metal could be enhanced.

The rates of desulphurisation were quite high and could theoretically be made considerably higher for the same air flow by more intimate mixing. This process could in practice only profitably be performed in the absence of the molten metal. The fact that the passage of cold gas through the melt resulted in considerable cooling might be partially overcome by the use of preheated air or the introduction of a fuel such as blast furnace gas or carbon into the air stream.

A possible development of post-blast furnace desulphurisation of molten iron might be:—

- (1) Blast furnace slag poured into a carbon lined reaction vessel and partially desulphurised by an air-fuel blow;
- (2) Molten iron poured into the slag and desulphurised by agitation;
- (3) The slag regenerated by a second air-fuel blow after separation of the iron.

Mr. Shanahan concluded by stating that an investigation was being made into the possibility of producing a liquid slag very rich in calcium carbide, which would be a very potent desulphuriser. Unfortunately, no experimental data were yet available.

## Discussion

In the course of the discussion on Mr. Shanahan's paper, it was suggested that desulphurisation of hot metal presented no problem but that consideration might be given to the elimination of silicon and phosphorus. In the open hearth furnace the sulphur from the pig iron accounted for only 23% or 24% of the sulphur in the system, the remainder coming from the fuel and other additions. Mr. Shanahan said there seemed to be a value below which there was little point in reducing the sulphur before the open hearth process, since extremely low sulphur iron appeared to take up sulphur more readily, from the fuel. Nevertheless, the special desulphurising processes such as the Kalling and Heuer could prove very valuable as they could be used to reduce a portion of the molten iron to a very low sulphur content and thus by dilution give a reasonably low average sulphur content.

Replying to suggestions that it might be more economic to remove phosphorus from the molten iron before supplying to the open hearth, he said that molten iron containing considerable carbon had very little oxygen potential available for appreciable oxidation of phosphorus. Chemical considerations showed that phosphorus removal was more effectively carried out in the open hearth when the carbon content of the iron had been oxidised to a lower level than that found in pig iron.

## Experiments on Feeder Heads

The second session of the conference began with a paper by Mr. W. H. Glaisher (British Iron and Steel Research Association) on "Experiments on Feeder Head Materials and Shape." Work on this subject aimed to improve the yield of ingots with which feeder heads were used, and to decrease the cost of such heads. The present paper was concerned with yield.

The more effective the means employed to retain heat in the head, the smaller the head needed to be. Various methods had been examined, many of them practicable but uneconomic. Commercial practice had concentrated on the use of firebrick or "compo" lined heads, sometimes with a feeding compound.

Heat could be lost from the top, the sides and the bottom of feeder heads. Heat loss from the top was examined by comparing unprotected ingots and ingots which had been protected at the top by Lepex, by vermiculite grains or by both together. When the heads were sectioned it was found that the main effect of the additions was to decrease or prevent bridge formation at the top of the head; the effect on the depth of the cavity was not very great. The heads examined were of ample size and the effect of such additions might, said Mr. Glaisher, be more important on other heads where there was a small safety margin.

Next, an attempt was made to decrease heat loss from the sides by the use of insulation. The materials examined were vermiculite brick (protected by a thin layer of cement) and two makes of high temperature insulating brick. Measurements of heat flow from the steel in the head to the refractory four inches from the bottom of the head, indicated that the total amount of heat taken from the steel at the point of measurement when using any of these materials was not much more than half that taken with the "compo" head. Secondly, most of the heat went to heat up the refractory itself.

The calculations showed that:—

- (1) If transfer to the walls could be cut to nearly half by the use of insulating materials, the total heat flow would be cut by about a fifth and the size of the head could be materially reduced;
- (2) The heat capacity of the refractory per unit of volume was at least as important, and possibly more important, than its thermal conductivity. The most important criterion for low heat capacity was low bulk density.

*Practical Application.*—Work had been carried out both on low density materials, and on thinner layers of material of more normal density, backed by an air gap or by insulating material, as a second means of ensuring low heat capacity.

Table I shows the results obtained when 3 in. thick high temperature insulating bricks were used in the head box of 24½-cwt. ingots, compared with the normal heads of rammed "compo" 1½ in. thick. The special heads had a volume, as filled, of about 8% of the ingot body against 13% for the normal heads. The ingots were rolled without cropping, the 2, 2½ or 3 in. billets from the top end then being cut back until a sulphur print showed a sound section. The results appeared promising, but the possibility of secondary pipe had yet to be examined.

TABLE I.

Steel	Discard From Special Head	Discard From Normal Head
2% Si Steel .....	9.0%	12.2%
1½% Ni-Mo Low Carbon Case-hardening Steel .....	6.2%	10.2%
1% Ni 1% Cr 0.2% Ca Steel .....	7.9%	11.2%

The insulating brick heads gave an average of 17 casts each, slight dressing being used when necessary, none being done until after the sixth cast.

Fig. 2 shows results obtained when thin, recessed refractory tiles were used. This method had been developed by Messrs. William Jessop and Sons Ltd. in co-operation with Messrs. Thomas Marshall and Co. Ltd. The gaps at the back of the recessed tiles were filled with vermiculite granules bonded with a small amount of Portland cement.

Recessed tiles had also been applied to a 4-ton ingot in which the refractory was inset in the top of the mould wall and remained in place throughout. To overcome a tendency to sagging, tiles with a honeycomb structure at the back were being tried and these were behaving well, though it was not yet possible to give any results on yields.

*Cost.*—Table II gives an estimate of material costs for the head of 35 cwt. ingots.

TABLE II.

Material	Cost per head	Life	Cost per ton
"Compo" .....	6d.	1	4d.
Vermiculite brick .....	15s.	1	8s. 6d.
H.T. insulating brick .....	16s.	say 5	1s. 9d.
Normal tiles .....	10s.	say 12	6d.
Recessed tiles .....	11s.	say 12	7d.

Mr. Glaisher said that these were very rough estimates and possibly on the high side for both the third and fifth items which might give longer lives than those quoted. He thought that with fairly small scale production of expensive alloy steels the use of the most efficient insulators should be economic, but as the rate of production rose, service life, ease of handling, and

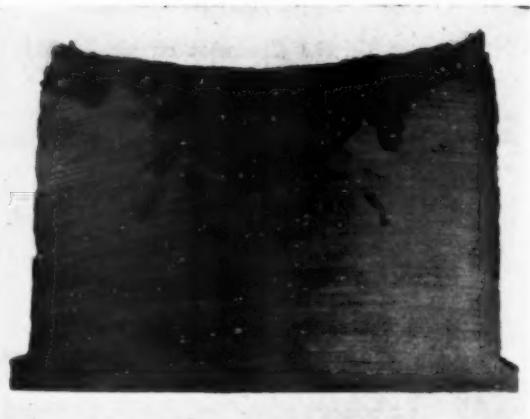


Fig. 2.—Feeder heads of high speed steel ingots (18.9% capacity.) Left—with solid tiles; right—with recessed tiles.

general convenience became increasingly important and might override other considerations.

*Shape of Heads.*—An experiment had been carried out to observe the effect of alteration in the dimensions of ingot heads without any change in volume. At the same time the opportunity was taken to compare the feeding obtained with "compo" lined heads, initially 15% of the total ingot weight, with that obtained with vermiculite lined heads, initially 10% of the total ingot weight. The experiment was carried out on 1-ton ingots of 0.5% carbon steel. The figures shown in Table III are for the weight of steel above the level of the base of the pipe, whether in ingot or head.

TABLE III.—DISCARDS DUE TO PIPE.

Ingot No.	Refractory	Height/Base Width Ratio	Minimum Possible Discard %
1	"Compo"	0.55	11.7
2	"	0.81	10.1
3	"	1.16	9.5
4	"	1.52	8.0
5	Vermiculite brick	0.57	14.7
6	"	0.84	13.3
7	"	1.18	7.1*
8	"	1.54	4.8*

\* Piped into ingot.

The experiments showed a possible difference in yield of about 3% between the shortest and tallest heads. Practical considerations were not discussed, such as the increased step at the shoulder which was produced with a tall, narrow head. This called for further research.

### Discussion

In the discussion on Mr. Glaisher's paper the usefulness of the work was generally agreed. Similar developments at two works were described by speakers. At one works 50-cwt., 12-sided moulds had originally had inserted tiles for feeder heads. Superimposed heads on the cut-down moulds were next tried but resulted in hanger cracks and bad surfaces on the head itself. A method was then developed whereby a truncated cone of sheet steel was used with the broad end just inside the top of the mould, and the space between it and the head-box filled with sand. This had had good results. In reply to a question, the speaker said that the cost worked out at 3s. 6d. per ton, which was more than covered by the smaller discard made possible. Another speaker said that in their normal head they had previously to discard

20% to 22% by weight of the ingot which was halved when recessed tiles were used. The use of a hot faced insulating brick, 1½ bricks thick, cut the discard to 8%.

Several speakers in discussing "short thick" and "long thin" designs of feeder heads expressed anxiety that secondary pipe might develop when the latter were used and Mr. Glaisher agreed that more work on this subject was very desirable. Other speakers said that the final criterion in judging feeder heads was the effect on yield at the rolling mills, a point with which Mr. Glaisher concurred but he pointed out that such measurements were not always easy to obtain, and the depth of pipe was the only convenient measurement readily attainable.

### Basic Refractories in Ladles

Mr. C. H. BACON of John Summers and Sons Ltd. then presented a paper on "Trials with Basic Refractories in Open Hearth Furnace Ladles." Almost all the steel, he said, at his works was low carbon rimming steel with high iron content slags, which were extremely severe on the usual type of ladle lining.

*Ladle Linings.*—Preliminary trials with panels of basic bricks, six bricks wide for the full height of the ladle, gave quite promising results with stabilised dolomite. When a complete ladle, except for the bottom, was lined with this material slag started to build up on the face of the bricks on the first charge and after the second charge it was evident that some skull had formed. In the process of removing this, it was found that the whole slag layer had to be removed, and a thickness of about ½ in. of brick was detached with it. Behind this, there was a further ½ in. to 1 in. of brick in a weak and crumbly condition. The ladle lasted a further four charges. A second ladle worked for seven charges before it became necessary to remove the slag layer. This had to be repeated after another five charges. It then worked for another three charges making a total life of fifteen charges.

Trials were also made with magnesium silicate refractories, at first with Serpex (serpentine enriched with magnesia) and subsequently with serpentine, with increasing proportions of first china clay and later fireclay being added to the latter.

A complete ladle lining bricked with chemically

bonded Serpex bricks, behaved in a very similar manner to the stabilised dolomite lining. The slag layer had to be removed on two occasions and some patching done to the brickwork. The total life was thirteen charges. Serpentine bricks with proportions of china clay and fireclay varying up to 30% were tested in panel trials. The tendency to build up slag persisted.

Generally, it was concluded that though a longer life could be gained from a ladle lined with basic bricks, this was not sufficient to offset the additional cost of the bricks, together with the maintenance that the ladles required during service. The chief difficulty encountered was due to the build-up of slag. It appeared that the action of basic slag upon a basic brick produced a viscous slag which tended to freeze on to the brick face, and until some means was found to overcome this slag build-up it did not appear possible to make a practical proposition of a basic lining for ladles.

*Stopper Rod Sleeves.*—In contrast to the inconclusive results of the trials on ladle linings, trials of stabilised dolomite sleeves on stopper rods had been successful. They had now been standard in one shop at the speaker's works for about eight years. Failure of a rod through sleeve wear was practically unknown, though it had previously been fairly common.

It had been found that wear on dolomite sleeves was negligible in the slag layer. The material was eroded in the metal layer but not as much as fireclay. Where a dolomite sleeve was in contact with, and immediately above, a fireclay sleeve it was subject to increased wear. A number of trials in which the height of this junction was altered indicated that the best results were obtained when it was just below the slag/metal interface.

### Discussion

The comparative costs of the sleeves used were commented on during the discussion on Mr. Bacon's paper, and Mr. Bacon said that the stabilised dolomite sleeves cost approximately three times as much as firebrick sleeves. In reply to other questions he said that they had had no trouble with thermal shock and he "admitted" that the jointing material for stabilised dolomite sleeves was ganister, but that no trouble had been experienced at the joints.

An interesting point made by one contributor to the discussion was that ladle life cost at his works was about two shillings per ton, which did not itself offer a great deal of scope for economy on basic ladles. However, if they could show a quality advantage by using the ladles which prevented rephosphorisation such a step would be worth while. Trials at these works had encountered difficulties with jointing materials when serpentine was being used. The jointing material of 20% serpentine and 80% magnesite, however, was giving promising results.

Another speaker said that longer lives had been obtained by the use of high alumina (54%) bricks. These were successfully used in a ladle in which steel was held for some time. Mr. Bacon said that they had obtained conflicting results with high alumina bricks and he was glad to know that a joint committee of B.I.S.R.A. and the British Ceramic Research Association was to look into the matter.

### Effect of Teeming Speed on Ingot Quality

The proceedings of the conference were brought to a close by the presentation and discussion of a paper on

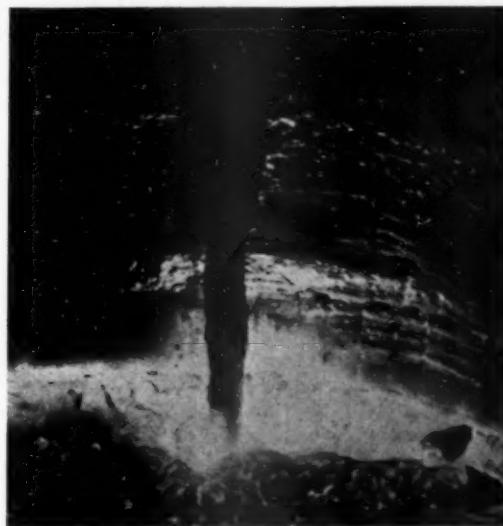


Fig. 3.—Stopper rod with stabilised dolomite sleeves worn thin during teeming.

"The Relation of Teeming Speed and Ingot Quality in Structural Steels," by Dr. L. Reeve of Appleby-Frodingham Steel Company, who described his paper as a brief summary of existing knowledge, together with an indication of present lines of investigation.

Dealing first with teeming rate, which in his works was controlled by nozzle size and not normally by throttling, he said that mould filling time, from beginning to end of a cast for any given nozzle size, went through a distinctive pattern. It first decreased as the nozzle wore and then increased again as the head of metal in the ladle fell.

The influence of mould size upon teeming rate as expressed in inches rise per minute had some important effects as the larger mould for a given ladle and nozzle size resulted in a slower rate of rise of steel up the mould.

The influence of teeming rate upon quality was related almost certainly to the properties of the initial skin of the ingot, and the thickness which had solidified by the time the mould was filled. In general, the more quickly the mould filled, the thinner this initial solid skin, and the greater the danger of ingot cracking. This was accentuated by hot steel and high sulphur steel, and killed steels were usually worse than balanced steels in this aspect. Cracking was reduced by slower teeming, but steel teemed too slowly might suffer from surface troubles due to lapping and shell, particularly if teemed too cold.

In balanced and rimming steels, teeming rate had an effect upon ingot skin thickness, by which was meant the solid steel nearest to the ingot surface. In general slower teeming rates and bottom pouring resulted in thicker skins, the precise effect on balanced steels depending on the degree of "balance."

A national survey of balanced steel ingot skin structures was now in progress. Dr. Reeve recalled that he had reported at previous conferences that such steels covered a fairly wide range of skin thicknesses. By taking advantage of the fact that several ladles are tapped from each heat of steel from large tilting furnaces,

it had been possible to investigate the influence of teeming rate upon medium to thin skinned balanced steels. 1-in. and 1½-in. nozzles were used. It appeared that slower teeming tended to thicken the skin of medium thick skinned steels but the influence of teeming time on the skin thickness of very thin skinned balanced steels—produced by a greater degree of ladle deoxidation—was negligible. The extremely thin skin suggested an almost complete suppression of rimming action, which could not be influenced by slowing up of the teeming rate.

A statistical survey of 216 casts of balanced steel which had been sampled for ingot corner structure at Appleby-Frodgingham had confirmed that small nozzles resulted in thicker skins and longer blowholes than larger nozzles. The influence of such changes in skin thickness on steel quality had not been quite so clearly established, though there was little doubt that they were related. In the case of the smaller 4-ton and 6-ton ingots used for structural sections, it appeared that large nozzles produced more defectives, particularly when in conjunction with very hot (or very cold) steel, and in particular when sulphurs were high.

In the case of the more complex factors which were

responsible for the surface quality of balanced steel plates, the position was not so clear, though generally Dr. Reeve believed that too large nozzles were undesirable and should rarely exceed 1½ in. for plate ingots.

#### Discussion

Discussion on Dr. Reeve's paper turned largely on the correlation between blowholes or cracks and various teeming factors. It was generally agreed that the rate of teeming was of importance. The weakening effect of sulphur on the high temperature properties of steel was also thought to have an effect, though one speaker vigorously denied from his own experience that sulphur had any effect whatsoever. The large size of mould and the low sulphur content of most American steels were suggested as contributory factors to the high teeming speeds common in the United States, and the great attention paid there to pouring temperature was also commented on. Dr. Reeve said that the Committee was not yet in a position to judge whether there was a correlation between blow holes and the amount of iron in the slag when tapped. He did not think there was any connection between the phosphorus content of the steel and the depth of blow holes.

## The Annealing and Heat Treatment of Ductile Cast Iron

*Continued from page 288*

and a low manganese content are desirable. But in order to obtain optimum impact resistance after annealing, some restriction should be placed on the upper limit of the silicon content. The authors suggest the following composition :

Carbon	2.9-3.2%
Silicon	2.0-2.3%
Manganese	less than 0.3%
Phosphorous	less than 0.05%
Residual Magnesium	not less than 0.05%

This is referred to in the text as the "preferred analysis" and ductile cast iron made to meet this specification has been found to give consistently good impact values after annealing.

The most satisfactory annealing process examined was the conventional treatment, consisting of soaking at 950° C. followed by slow cooling. The impact figures after sub-critical annealing were not quite as good, but with ductile cast irons of low manganese contents the results were still of the order to be expected from a good quality blackheart malleable iron.

Mechanical tests were made on a number of hardened and tempered ductile cast irons and typical results have been quoted. Under the conditions adopted in these tests there was a tendency for irons containing approximately 2% silicon to give lower tensile strengths than irons containing approximately 1½% silicon. This difference was found to be due to decomposition of the martensitic matrix in the higher silicon iron resulting in the random precipitation of a large number of small graphite particles.

The phenomenon has not been fully investigated by the authors but it was for this reason that castings to be

used in the hardened and tempered condition were made from irons having a lower silicon content than that preferred for castings to be used in the fully annealed condition.

#### Acknowledgments

The authors wish to thank the Directors of Leyland Motors, Limited for permission to publish this work and Mr. Walter West for his encouragement during its progress.

## A.P.V. to Make Stainless Steel Valves Under Licence

ACCORDING to a Mutual Security Agency announcement, the A.P.V. Company, Ltd., of Wandsworth Park, London, will use the processes and technical data of the Cooper Alloy Foundry Company, of Hillside, New Jersey, U.S.A., for the manufacture of stainless steel valves. The Cooper Company, which is a leading manufacturer of corrosion-resisting valves, will furnish designs, engineering and production drawings, and information on techniques and procedures for the manufacture and use of valves used in the processing of chemicals, brines, petroleum, explosives, paper and other products involving corrosive and hazardous liquids and gases. This data will be available under a 10-year licensing agreement, with royalties to be paid for all valves produced by Cooper processes and sold or used by A.P.V. during the period. The U.S. Government has agreed to convert into dollars any sterling royalty payments which the American firm is unable to convert through regular foreign exchange channels, up to a maximum of 153,500 dollars over the 10-year period. A.P.V. sales of stainless steel valves are expected to reach a volume of from £100,000 to £150,000, with exports yielding the United Kingdom valuable hard currency through A.P.V.'s extensive overseas organisation.

# A Modern Heat Treatment Plant

## Wild-Barfield Installations at Blaw-Knox, Ltd.

THE days when it was considered natural that the heat treatment shop should resemble a cross between Dante's Inferno and the Black Hole of Calcutta are happily past, but there are many heat treatment engineers who, having inherited a shop initially of that type, have to be satisfied with a compromise which falls short of the ideal. They will doubtless be envious of the engineer in charge of the Blaw-Knox heat treatment shop at Rochester, which was built for the sole purpose of housing a complete heat treatment installation. The objectives were that it should be light, roomy and laid out with equipment that would give the greatest flexibility. Dust, noise and smell were to be eliminated, and a flow of work was to progress through the processes in the shortest possible time and with the minimum of handling. That these objectives were attained is due in no small measure to the planning and foresight of both Blaw-Knox and Wild-Barfield, who supplied the equipment and were brought into consultation at an early stage.

The components treated include a range of gears, pinions and shafts for Blaw-Knox scrapers, motor graders, excavators, concrete pumps, winches and mixers, in a variety of case hardening steels from En 32 to En 39, and the general purpose of the equipment installed is the gas carburising of such parts, together with other treatments such as refining, normalising, hardening and tempering.

For carburising there is a Wild-Barfield gas carburising



A general view of the heat treatment shop showing, from left to right, degreaser, vertical forced air circulation furnace, oil quench tank, water quench tank, unloading pit, cooling pits each side of the gas carburising furnace, and loading pit.

furnace Model No. G.C.F. 2448, with three heavyweight retorts and two cooling pits. This furnace is of the vertical type, having a work chamber of 24 in. diameter by 48 in. deep, and is fitted with heavy hairpin elements of nickel-chromium alloy giving a rating of 100 kW. The maximum operating temperature is 1,000° C.

Prepared town's gas is used for carburising, having as active carburising agents methane and carbon monoxide, which are contaminated in the raw town's gas by carbon dioxide, oxygen, water vapour and small amounts of organic sulphur compounds which make it unsuitable for carburising without treatment. At Rochester the raw gas passes through a Holmes Catalytic Sulphur Removal Plant, where 80-90% of the total organic sulphur compounds are reduced to hydrogen sulphide and removed by activated bog ore. This gas—substantially free from organic sulphur compounds—then passes into the Wild-Barfield gas preparation unit (Model Mark II, G.C. 300), where it passes over a catalyst at 900° C., through a non-contact type cooler-condenser and, finally, through silica gel driers. The gas then has the following approximate analysis: unsaturated hydrocarbons—1.5%; carbon monoxide—21%; methane—17%; hydrogen—51.5%; nitrogen—remainder. This gas has the ability to carburise at any given temperature at almost the maximum theoretical rate, and shows little or no tendency to sootting, even at high rates of flow.



A view from the opposite end of the shop illustrating, on the right, the horizontal "hairpin" furnace, loading trolley and charge progress recorder.



The purged retort entering the gas carburising furnace and, in the background, the gas preparation unit and catalytic sulphur removal plant.

Refining, normalising and hardening operations are carried out with a Wild-Barfield horizontal "hairpin" furnace Model No. H.H. 4018, fed with a controlled atmosphere from a G.W.B. burnt town's gas plant Model No. A.P. 500, which has a capacity of 500 cu. ft./hr. and is provided with silica gel driers. This furnace has a chamber of grooved refractory bricks giving a working space of 40 in. wide by 18 in. high by 56 in. deep. The elements are of nickel-chromium alloy rods of hairpin shape for easy removal and a hearth tray of heat-resisting material protects the floor elements and brick-work. 1,050° C. is the maximum operating temperature, and the furnace is used for subsequent refining and hardening after gas carburising. It is also used without atmosphere for the normalising of rough forgings, before machining for gas carburising, and the hardening, with atmosphere, of carbon and alloy steels.

Two quench tanks are provided, one for water and the other for oil, the latter using Shell-Wild-Barfield No. 1 quenching oil which is cooled by means of a Serek oil cooler.

After hardening, the parts are tempered in a Wild-Barfield vertical tempering furnace Model No. 3232 C. having a removable work basket of 32 in. diameter by 32 in. deep. This furnace works on the forced air circulation principle, having a fan at the bottom of the chamber which circulates the hot air around the work placed in the work basket. Its maximum operating temperature is 700° C. and it is also used for general purpose heat treatments below its maximum operating temperature.

A further vertical forced air circulation furnace, Model No. T.R.T. 1010, with a 10 in. diameter by 10 in.

deep basket is provided for regenerating the silica gel from the gas preparation unit, and for tempering parts within its capacity.

Where applicable these equipments are supplied with the necessary transformers, contactors, controllers, charge progress recorders and single point recorders, and a Vickers diamond pyramid hardness tester is provided for the test laboratory set adjacent to the heat-treatment shop.

The layout was planned for an easy flow of work through the various stages, as will be seen from the illustrations. An overhead crane traverses the line of furnaces, cooling pits and quench tanks, while work for the horizontal "hairpin" furnace, on the opposite side of the shop to the gas carburising plant, is fed into the chamber by means of a trolley having a roller table for sliding the trays into and out of the chamber.

The plant is coping with practically the whole range of carburising steels from En 32 to En 39, and experience has shown that this flexibility, taking into account also the varying sizes and weights of the components, is combined with the precise metallurgical control exercised regarding the depth and type of case. In all work treated, the case is entirely free from hyper-eutectoid features, and use is made at all times of a controlled atmosphere in order to avoid scaling and the consequent necessity for shot blasting or pickling.

### Tinplate Output Record

THE new five-stand cold reduction mill at the Trostre Works of the Steel Company of Wales, which started operation only seven months ago has already begun to indicate its productive capabilities. Last month, during an eight-hour shift, the mill rolled 452½ tons of steel strip into thin tinplate gauges—twice as much as has ever been rolled before in this country in the same period. The mill, which is capable of rolling at a speed of 4,500 ft./min., normally operates at 3,000 ft./min., and during this record shift it rolled at a rate of 9,000 tons per week, which represents 180,000 boxes.

A significant fact is that the men who operate this mill have all been drawn from old-type tinplate works in South Wales, and without exception none of them has ever before worked on a continuous cold reduction mill—indeed, many of them have never before even seen a mill of this type. They have been trained at the Trostre Works on the mill itself since production began last October, and the speed with which they have mastered the new methods is ample evidence of the adaptability of the South Wales tinplate worker.

Other departments in the new works are keeping in line with the achievements of this mill as is shown by the fact that the continuous pickle line recently processed 456 tons during an eight-hour shift.

### Change of Address

WE are advised by Fletcher Miller, Ltd., makers of cutting oils and industrial lubricants, that their Southern Works has now been transferred from Hayes, Middlesex. The new address is: Alma Wharf, Dormay Street (off Armoury Way), Wandsworth, London, S.W.18. Telephone: Vandyke 6033/4. Telegrams: Kooledge, Put., London. All communications regarding supplies and deliveries within the London and Home Counties area should be sent to the Wandsworth address.

# The Influence of Shot Peening on the Fatigue Properties of Steel

By W. J. Harris, B.Sc., A.Inst.P.

(Chief Physicist, Central Laboratory, De Havilland Aircraft Co. Ltd.)

*Recent years have seen increasing use made of shot peening for the purpose of improving the resistance of components to failure by fatigue. Beneficial results may be obtained in cases where—due to adverse residual stresses of metallurgical origin, or to geometrical features likely to result in stress concentration—the endurance ratio is below 0.45–0.50.*

THE insidious nature of fatigue failure and the complexity of the design problems in cases where combinations of adverse vibratory stresses, stress concentration, corrosion, etc., arise, make any means of producing an improvement in the fatigue properties of the steel used worthy of the most searching investigations.

A study of the work of Almen<sup>1</sup> and his collaborators in the United States indicates the importance attached to such investigations, and the encouraging results of their work have been mainly responsible for the impetus given to shot peening in this country. Although there is a wealth of American data on the shot peening of components and test bars, the work has been mainly directed towards the solution of specific problems, and the broad fundamental principles of the mechanism involved in producing an improvement in fatigue properties have not been deduced from this data.

Our own work, carried out on a very modest scale, has also been directed with certain production problems in mind, but an attempt has been made to resolve certain anomalous results found in American data, and to complete a critical survey of shot peening in relation to fatigue from which certain fundamental principles could be deduced.

## Experimental Results and their Interpretation

In the following account, frequent reference will be made to the endurance ratio  $r$  which is defined as

$$r = \frac{\text{Semi-range of Nominal Fibre Fatigue Stress}}{\text{Ultimate Tensile Strength of Surface Fibres}}$$

The emphasis on surface fibre stresses is significant, as it constitutes the basis of the subsequent interpretation of the experimental results. The ultimate tensile strength (U.T.S.) has its usual definition, and in the cases discussed relates to values obtained from tensile tests or deduced from surface hardness measurements. The basis for the endurance life in all the work described below is  $10^7$  cycles, unless otherwise stated.

The results of experiments to determine the effect of shot peening on the fatigue strength of a number of steels are presented below.

## Medium and High Carbon Steel

Fatigue tests on three different gauges of D.T.D. 5A patented carbon spring steel wire were carried out on a Haigh-Robertson Wire Fatigue Machine, and the

endurance ratios in the 'as drawn' and 'shot-peened and tempered' conditions are shown in Table I.

TABLE I.—ENDURANCE RATIOS ON D.T.D. 5A STEEL WIRE

Gauge (S.W.G.)	Endurance Ratio ( $r$ )		Improvement %
	As Drawn	Shot Peened and Tempered (300°C., 1 hour)	
7½	0.243	0.360	48.5
9	0.320	0.366	17.2
10	0.294	0.332	13.0

The shot peening conditions were chosen quite arbitrarily and, consequently, the optimum peening has not been applied to bring the endurance ratios up to the neighbourhood of 0.45–0.50, but it is maintained that this is possible, and in the case of the 7½ S.W.G. wire quoted, a 100% improvement could be achieved.

Where the tempering operation precedes the shot peening, a further improvement results, but the effect of tempering is not of the same magnitude as that of shot peening.

It will be seen from the above results that the 'as drawn' endurance ratios are 'defective,' since for this steel they can be raised to the 0.45–0.50 range by suitably hardening and tempering. Fatigue tests on hardened and tempered D.T.D.5A were not carried out, but the results of Lea<sup>2</sup> on a 0.62% carbon steel indicate the probable trend. Lea found that with such a steel at a U.T.S. of 80.4 tons/sq.in., the fatigue limit was  $\pm 20.1$  tons/sq.in. in the cold drawn condition, and  $\pm 39.4$  tons/sq.in. in the hardened and tempered condition, the respective endurance ratios being 0.25 and 0.49.

It will be noted that hardening and tempering produces almost 100% improvement in fatigue properties, as compared with the cold drawn condition, and that the endurance ratio of 0.49 is virtually the maximum.

The reason for the 'defective' endurance ratio of 0.25 in the cold drawn condition lies in the condition of the surface fibres, and although it is beyond the scope of these notes to theorise on the metallurgical reasons for the 'defective' cold drawn surface, the suggestion that it is due to the existence of residual tensile stresses in the surface after cold drawing appears likely, bearing in mind the nullifying effect of induced compressive stresses due to shot peening.

It will be appreciated, therefore, that where the maximum endurance ratio has already been attained, e.g. by suitable heat treatment, shot peening would not

1. The Effect of Shot Peening on the Mechanical Properties of Steel. (Na-115) Metallurgy Division (Div. 18) N.D.R.C.

2. Prevention of the Failure of Metals under Repeated Stress. Battelle Memorial Inst.

significantly improve the fatigue properties. The negative results we have obtained on hardened and tempered S.11 steel confirm this view, as do some American data<sup>3</sup> from tests on X4340 in the 65 tons/sq.in. U.T.S. condition. The fatigue limits for super-finished and shot-peened conditions were  $\pm 31.3$  and  $\pm 30.8$  tons/sq.in. respectively and the endurance ratios 0.482 and 0.475.

### Case-hardened Steels

Bearing in mind that we are mainly concerned with fibre stressing, the following data indicate that case-hardened steels, finish ground to production standards, fall into the class of steel having 'defective' endurance ratios.

For example, S.15 steel carburised and heat treated in an orthodox manner had a surface hardness of 754.5 V.P.N., equivalent to a U.T.S. of 146 tons/sq.in., and a Wohler fatigue limit (10<sup>7</sup> cycles) of  $\pm 46$  tons/sq.in. This corresponds to an endurance ratio of 0.315.

American data<sup>4</sup> on pack-carburised SAE.4820 steel show similar features. 'Burnt ground' specimens have their fatigue resistance improved by shot peening from  $\pm 44.5$  tons/sq.in. to  $\pm 62.5$  tons/sq.in., which is the same as is achieved by super-finishing. This value indicates a high order of improvement, but the theoretical maximum (probably  $\pm 70$ –73 tons/sq.in.) has not been attained.

The condition 'burnt ground' indicating unsatisfactory grinding, would appear, however, to be little inferior to that achieved by normal production grinding when the value of  $\pm 44.5$  tons/sq.in. is compared with the  $\pm 46$  tons/sq.in. obtained in the S.15 tests.

From the foregoing, shot peening would be expected to show a minimum of 40% increase in fatigue properties on normal production ground case-hardened surfaces.

### Tensile Strength and the Endurance Ratio

It is logical to enquire, on accepting the fact that 'defective' endurance ratios can arise from fabricating and processing techniques, whether there are not metallurgical conditions of the steel achieved by, say, heat treatment which could have the same result.

To answer this question, a series of fatigue tests was carried out on S.11 steel test pieces which had been heat treated to give U.T.S. values in the range 48.9 tons/sq.in. to 128.8 tons/sq.in. The fatigue test results on the plain heat treated specimens, and on those shot-peened, showed no significant departure from an endurance ratio of approximately 0.46.

These results indicate that, over the tensile range investigated, the maximum endurance ratio has been reached, and no significant improvement results by shot peening. The static mechanical properties do not play a significant role in our interpretation of the shot-peening mechanism, only the endurance ratios.

### Stress Concentration

Any review of the relation of fatigue properties to design requirements is incomplete without a consideration of the effect of stress concentration factors involved in the use on components of threads, keyways, holes and abrupt changes of section, etc. The satisfactory functioning of many components can be critically dependent on the geometric form of the component,

which may be mechanically efficient but vulnerable to fatigue due to the adverse effects of stress concentration. The mitigation of stress concentration factors becomes of paramount importance, therefore, in order to prolong the service life of components, and to realise to the full the ingenuity of design.

From our own work on shot peening notched fatigue bars, and from certain American data, it appears that, with the correct peening conditions, stress concentration factors can be completely eliminated with the restoration of parallel bar properties, as can be seen from the results shown in Table II of fatigue tests on S.11 steel bars heat treated to 59.9 tons/sq.in. U.T.S. and machined with a semi-circular circumferential notch of radius 1/32 in. Shot peening has conclusively restored the parallel bar properties with  $r = 0.470 - 0.472$ , and has eliminated the stress concentration factor of the notch.

TABLE II.—RESULTS OF NOTCH FATIGUE TESTS ON S.11 STEEL BARS

Condition	Fatigue Limit ± tons/sq.in.	Endurance Ratio
(1) Turned and polished notch ..	17.28	0.288
(2) Turned notch .. ..	17.19	0.287
(3) As (1) and shot peened .. ..	28.25	0.472
(4) As (2) and shot peened .. ..	28.04	0.470

Information as to the complete effectiveness of shot peening is obviously more difficult to obtain on components than on test bars, owing to the lack of test equipment capable of accommodating large components. In consequence, a good deal of the evidence to support the efficiency of shot peening in mitigating stress concentration effects in large components must derive from actual service performance and, particularly, the incidence of fatigue failures.

American data<sup>5</sup> indicate the great improvement in fatigue life of steel components due to shot peening, and one example may be quoted in which an increase of 40% in the fatigue life of propeller hubs (Steel : SAE.3140) was achieved by shot peening normal production-finished hubs.

Our own experience on components is confined to service data on certain propeller components. Fatigue cracking in the radius of spider arms seems to have been effectively cured by shot peening. On the two-piece propeller barrel, the bolt hole bosses were subject to fatigue cracking according to American information, and shot peening has eliminated the trouble. Although no actual cracking has been experienced on our split barrels, the shot peening is being applied in the light of American experience.

### Conclusion

Although the foregoing survey of the effect of shot peening on fatigue is by no means comprehensive, it is obvious that shot peening can be a means of increasing the fatigue resistance in certain circumstances. Among the cases where beneficial results are obtained may be included :—

- (1) Steels showing endurance ratios below 0.45–0.50;
- (2) Components whose design includes features likely to result in stress concentration effects;
- (3) Components featuring adverse residual stresses of metallurgical origin, such as the junction weakness in fatigue after induction hardening, and residual stresses in certain types of electroplated coatings.

3. (Na-115) Part I, Vol. 1, Case 13.

4. (Na-115) Part I, Vol. 1, Case 15.

5. (Na-115) Part II, Vol. 2, Case 42.

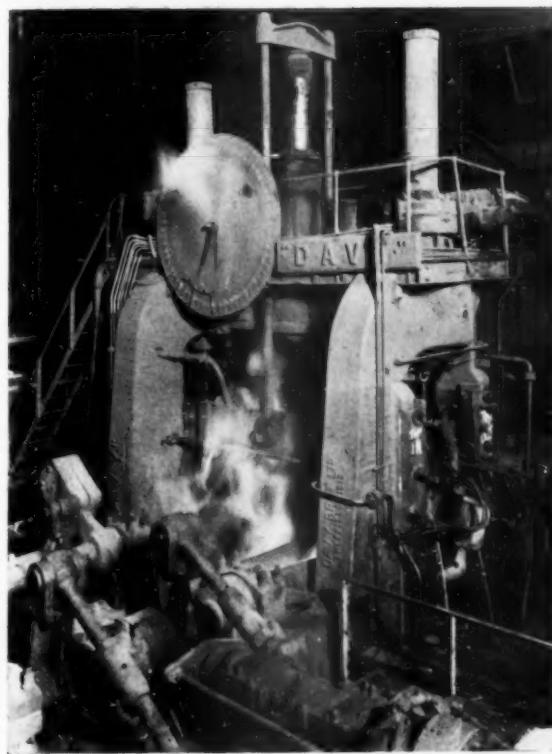
# Automatic Lubricator for Rolling Mills

## Wakefield Installation at Scunthorpe

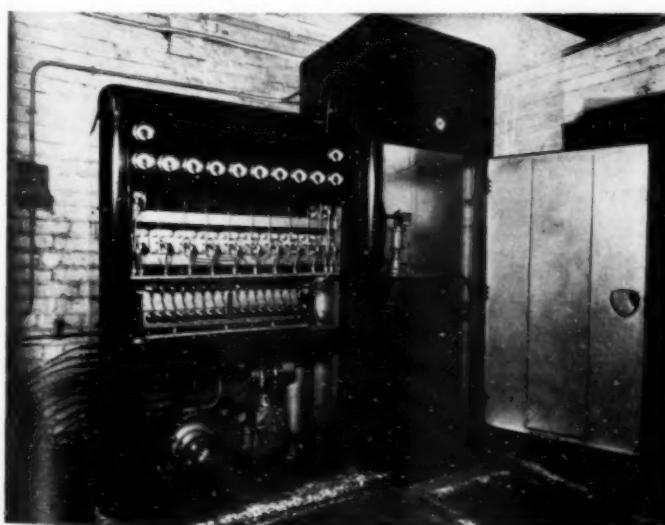
To meet the precise lubrication needs of large plant such as rolling mills in steelworks, C. C. Wakefield and Company have introduced an automatic greasing lubrication battery of an improved, up-to-date design. The first model to be built on the basis of data obtained from a strenuously tested prototype has been operating at the Appleby-Frodingham Steel Company, Scunthorpe, since November, 1951, where it has been responsible for the lubrication of the main roll bearings and breast rolls of their 42 in. Davy reversing slabbing mill.

This new Wakefield "Roll Mill" Lubricator is unusually compact: although the equipment is as comprehensive as may be expected, it is completely housed in a steel cabinet roughly the same size as an average wardrobe. Appleby-Frodingham engineers carried out the actual installation.

Although entirely new in layout, the design and operation of the lubricator is very straightforward. Both the front and back panels and door are completely



Grease is supplied to the main roll bearings and breast rolls of this 42 in. Davy reversing slabbing mill by the Wakefield lubricator. Six of the feed lines can be seen at the top left-hand side of the mill.



The Wakefield "Roll Mill" Lubricator. The compressor and grease outlets can be seen, lower left, whilst the middle left is occupied by the oil pump, feed regulators, relief valves, pressure cylinders and bleed valves. Lubricant pressure gauges are located top left, and on the right are the barrel and primer pumps.

removable for easy maintenance, and may be locked in place upon reassembly. The principle of operation is by a well-proven method: every bearing is fed with grease by a piston which is actuated by oil under pressure. Grease output is claimed to be ample for all industrial purposes, and there is an automatic alarm and safety system should the pressure in any of the grease lines exceed the permissible limits.

The internal arrangement comprises:—

- (a) Fully automatic electric air compressor.
- (b) Air-operated grease primer pump.
- (c) Multiple feed, motor driven oil pump.
- (d) Pressure cylinder, one per feed (12).

Pressure gauges, switches, piping etc.

The grease feeds are all capable of being individually regulated from maximum to zero. As the lubricator is totally enclosed, the grease is transferred from original  $3\frac{1}{4}$  cwt. grease barrels to the terminal points without manual handling or exposure to the atmosphere: the average consumption of grease is  $2\frac{1}{4}$  barrels per week. Unlike the usual grease lubricators, there are no wearing

parts likely to be affected by foreign particles in the grease. All the main pipes are 1 in. diameter with a maximum length of 65 ft. and a minimum of 25 ft.

For the lubrication of the main roll bearings there are eight feeds—two top and bottom for each side; for the breast rolls there are four feeds—two each side; this makes a total of twelve points.

When in operation, the compressor supplies air to the primer pump, which in turn delivers the grease from the grease barrel to the rear ends of all the pressure cylinders. The multiple oil pump then feeds oil, under pressure, to the front, or opposite ends of the pressure cylinders: through the medium of the hydraulic-type pistons inside the cylinders, this forces the grease out to the grease outlets, also under pressure. The pressure developed in each feed (maximum 1,500 lb./sq.in.) is registered on individual gauges incorporated in the top panel of the cabinet.

The oil pump is driven by a fractional H.P. geared motor at a speed of 25 r.p.m. At full feed this ensures

a grease output from each cylinder of 1 lb. 3 oz./hour. This quantity can be reduced by making use of the regulators incorporated in each pump unit, because any variation in the quantity of oil pumped by the pump will effect a similar modification in the amount of grease fed from the pressure cylinders.

When the grease in the pressure cylinders has been totally expelled, the cylinders are automatically recharged with grease by means of a totally enclosed valve gear contained in each cylinder. This operation takes only a few seconds and the feed is resumed forthwith. The oil used to expel the grease is returned automatically to the oil pump, thus making a closed circuit. Only very occasional topping-up is required.

Should a stoppage occur in any individual feed line causing an excessive pressure to be built up, an electric alarm bell operates and a relief valve reduces the pressure to safe limits. Suitable push-button switches with overload releases for the control of the air compressor and oil pump motors are fitted on an interior panel.

## All-Welded Dock Gate Launched

FROM the same slipway which saw the launch of many tank landing craft during the War, a somewhat different type of "vessel" was launched on Friday, 23rd May. This was one of a pair of all-welded dock gates which has been designed and built by Messrs. Head Wrightson & Company Limited for the Port of London Authority. The gates were fabricated at the Company's Teesdale Works and launched into the River Tees where tugs were waiting to tow them to the mouth of the river where a larger tug took over to continue the journey down the North Sea to the River Thames. The gates are for the King George V Dock and are to be interchangeable with any of the three positions of the 100 feet wide entrance lock. They will be stepped into position by the P.L.A. floating crane.

Head Wrightsons' are admirably situated for this type of work with their specially skilled men, and the facilities of first class constructional and fabricating shops near to the launching slipways. Further orders for eight gates have already been obtained for the London area, following the success of the installation of all welded gates at Calais by this Company. It is interesting to note that the weight of each leaf of this welded design is 240 tons against that of approximately 350 tons of the existing riveted type.

Each leaf is 58 ft. long  $\times$  49 ft. 6 in. deep  $\times$  7 ft. 3 in. mould width and consists of 17 plate ribs or decks, suitably stiffened where necessary. The skin plating is butt jointed and is composed of eight strakes on both the flat and curved sides. Vertical stiffness is provided by five plate diaphragms extending the full depth of the gate, the centre diaphragm being watertight for its full depth, the two outer being watertight between the top of the air chamber and the second lowest deck thus forming four tanks in the lower air chamber and two



The dock gate on the slipway

tanks in the upper tidal chamber of the gates. The space between the bottom and second decks is also tidal on the pressure side.

The heelpost and mitre post are built from plates, welded in the form of a channel and joist sections respectively, the heel, mitre and sill timbers being of greenheart. A central core plate with stiffener brackets at deck centres is welded directly to the web of the heel post.

Although the gates are of the buoyant tank type, rollers have been fitted to suit the existing roller paths. A crocodile arm, or lever, also of welded construction, is provided to operate the gate from the existing hydraulic machinery. Workmanship throughout has been under Lloyd's supervision and they also certified the hydraulic pressure tests on the tanks of the gate.

# Twenty-Five Years of Electric Furnace Manufacture



General view of the Birlec Works.

ALTHOUGH the culminating event of the Birlec Silver Jubilee celebrations, an excursion to Blackpool with the Company entertaining some 1,300 employees and friends, may be regarded as the highlight of the week's activities, a smaller function in the form of a Dinner to long-service employees at the Welcombe Hotel, Stratford-on-Avon, is of much greater significance in the light it throws on the spirit of the firm and the loyalty which it has attracted to itself. Although it is only twenty-five years old this month, and although twenty years ago it was still quite a small concern, the Company to-day has forty employees with a minimum of twenty years' service—a record of which it is justly proud.

## A. Glynne Lobley

When Birmingham Electric Furnaces Ltd. (the name was changed to Birlec Ltd. just after the war) was started in 1927 as an outlet for the nickel alloys of the

then parent company, Henry Wiggin and Co. Ltd., the payroll contained only four names: A. Glynne Lobley, head of the Company from its inception until his death in January 1950; George P. Tinker and T. G. Tanner, now Managing and Technical Directors, respectively, and E. Rawes, now Secretary to the Company. There can be no doubt of the important influence of Glynne Lobley on the development of Birlec to what is probably the largest firm in the world engaged in the manufacture of electric furnaces and allied equipment. He is remembered by his former colleagues, not only as a man of technical brilliance, a capable business man and a just administrator, but also as a kind man and a good friend. In reviewing the Company's progress at a Press Luncheon last month, Mr. J. H. Crossley, Sales Director, emphasised the advantages of having a metallurgist and scientist as chief. Mr. Crossley went on: "He (Glynne Lobley) had a brilliant technical mind, and a character—stern if you like, meticulous certainly—which refused to be daunted. With that combination of qualities he solved many problems and we can count to our credit many pioneering designs which, after first appearing hopeless, were by persistence solved. I could mention many types but will confine myself to three; the rotary drum annealing furnace—over 200 of which were produced in war-time and in which the vast majority of cartridge cases were annealed, not only in Great Britain, but throughout the Empire; the duplex nitrider for aero-engine crankshafts and liners—a similar number of which were made—after one of our first orders had been cancelled because, in accordance with the best German industrial opinion, our design would never work; and thirdly, our malleable annealing equipment—now spreading round the world—and which is, mercifully, I am pleased to say, a furnace used mainly for peacetime purposes."

## Nickel Associations

Mention was made earlier of the fact that Birlec originated as a new outlet for the



The Research Department's furnace demonstration section.



View of the plating shop

nickel alloys of Henry Wiggin and Co. Ltd. To-day, the two are sister firms in The Mond Nickel Co. Ltd. and are, thereby, part of The International Nickel Company of Canada—the largest producer of nickel in the world. In this way, Birlec is fortunate in that it is able to provide for its staff recreational and welfare facilities—including a non-contributory pension scheme—which might not otherwise be possible. In spite of the fact that the staff numbers almost 800, Birlec is still a firm of individuals as it is on the skill of individuals that the products depend. Lest Mr. Crossley's remarks quoted earlier should, when removed from their context, suggest that mass production is the keynote, it should be emphasised that the output consists primarily of tailor-made equipment, which calls for a considerable display of individual skill.

Originally located in small premises in George Street, Birmingham, the Company obtained its present 3½-acre site at Erdington in 1930. Although the first buildings, two small bays and an office block, looked isolated on the plot of land between the tram track and the Birmingham-Fazeley canal, it became evident even before the war that what had appeared in 1930 to be generous provision for future expansion was insufficient, and a concrete platform was thrown over the canal to form the floor of the largest bay—an impressive piece of constructional engineering. In addition, premises at 95, Tyburn Road, about half-a-mile from the Main Works, are occupied by the Birlec Heat Treatment Division, a small but important part of the organisation undertaking heat treatment work on a normal commercial basis.

#### Varied Products

Although there have been many digressions from the original aim of providing equipment to ensure efficient and economical heat treatment, this has remained the central and main theme of the Company's activities. Besides introducing completely new furnace

designs, Birlec have pioneered new heating applications in this country—continuous furnace copper brazing, continuous bright annealing, and gas carburising, to mention but three. Special attention has been paid to the metallurgical problems involved in the application of gaseous atmospheres, and to the mechanical problems in designing various types of conveyor equipment. Undoubtedly some of the greatest advances which have been made in heat treatment plant in recent years have concerned the means of handling the material to be treated.

One of the departures from the original object in starting the firm has been the manufacture of gas-fired equipment which, although using a large amount of nickel alloys, does not depend on their resistance properties for the generation of heat. A larger deviation has, however, been into the melting field where, on the whole, the best practices of American design have been followed in collaboration with American associates. A start was made with the

indirect arc type, then followed the direct arc units, the first being installed just before the war, and latterly, with the collaboration of Ajax-Northrup, high frequency induction units for steel and low frequency induction units for aluminium and other non-ferrous alloys.

Considerable attention has also been paid to the development of induction methods of heat treatment which are likely to be increasingly adopted in the future, particularly where large numbers of similar parts are involved. Not only in heat treatment is the induction method being used, but also for other heating operations such as those preceding forging.

The latest manufacturing interest is the Birlec Lectrodryer, a moisture remover based on activated alumina. Although this type of equipment is now of special application to the chemical and general engineering industries, it originated in a metallurgical application—the drying of the atmosphere in a bright annealing furnace.



The paint shop and test bay

# Reduce Costs and Increase Production by Preventive Maintenance

By Dr. D. F. Galloway, Wh.Sch., M.I.Mech.E., M.I.Prod.E., M.I.I.A..  
A.M.I.E.E., B.Sc.Hons., M.Inst.Pet.

*Director of the Production Engineering Research Association of Great Britain.*

*This article was prepared in connection with a Conference on Preventive Maintenance, organised by the Production Engineering Research Association, and held at Melton Mowbray at the end of May. The Conference, which was attended by more than 350 directors and managers of a wide range of manufacturing firms, is the first of a series being planned by the Association to provide industry with the most up-to-date information on aspects of industrial practice concerned with the speed and economy of manufacture.*

MANY engineering firms have progressively improved their manufacturing practice by carefully planning all stages of production. The success or failure of the methods used has been measured against the yardstick of speed and economy of production. Unfortunately, very few firms have anything like the same progressive approach towards efficiency in the maintenance of their production plant. This is often because executives do not realise that many of the technical difficulties which limit the speed and economy of production can be partially or even completely overcome by ensuring that machines and equipment function at their real maximum capacity with minimum delays due to failures, etc.

The cost of maintenance itself, the cost of lost time due to breakdowns and emergency repairs, and often greatest of all the loss due to the inefficiency of imperfect plant working below its maximum capacity constitute only three of the items which in a regrettably large number of factories are accepted as unavoidable and virtually irreducible constituents of overheads. Although in some companies the installation of preventive maintenance schemes has cut maintenance and repair costs by about 50%, this is generally of secondary importance to the direct gain in productivity arising from plant of all types being continuously in efficient working order.

The country's economic position demands that industry should abandon the odd-job outlook which characterises most of the maintenance work in Britain, and substitute for it rational schemes of preventive maintenance. The enormous burdens thrust upon the industry by the need to expand arms production, increase exports and maintain living standards at home can only be sustained if there is an all-round increase in technical efficiency. This task has recently been made even more difficult by the restriction of supplies of capital equipment to British firms, and under these circumstances especially, preventive maintenance is undoubtedly one of the most effective methods of securing the desired increase. The need for preventive maintenance is also greatly emphasized by the increasing mechanisation of manufacturing processes and the urgency of achieving continuous production at higher rates.

It is certain that a significant contribution to national recovery can be made by the wider application of preventive maintenance techniques in which plant

defects are detected before a breakdown occurs or repairs become costly. The principles of economy and efficiency should be applied in maintenance work no less than in production. It is just as necessary to combine the best skills, methods, tools and equipment to do the best work in the maintenance department as it is in the production department.

## Applicable to all Firms

Experience has proved that preventive maintenance under almost all conditions is economical maintenance. Hit and miss methods are always costly both in terms of machine breakdowns and lost production time. No firm is too small to benefit from a simple scheme of maintenance. Such measures as regular inspection and lubrication, and periodic minor adjustments and correction of faults, which are the principal features of preventive maintenance, can be applied in the smallest firm. The minimum requirement is that measures be applied to plant whose failure may seriously affect production or safety. A simple costing control system for maintenance is in itself a significant step to increased efficiency. One firm has reduced overall maintenance costs to three-quarters by arranging for a man to spend about half his time maintaining a two-card costing system.

## The Importance of Adequate Records

The use of an excessive number of forms has led to criticisms of preventive maintenance by practical men, but their criticisms are justified only in cases where there have been errors in planning, for no scheme for the control of preventive maintenance should be more complicated than the usual production control scheme. An interesting example of the way in which clerical work can be reduced in some cases is provided by the Douglas Aircraft Company, where mechanics patrol areas in which machine failures are most likely to occur. The majority of the firm's maintenance work is carried out in this way, and much of it is so inconsequential in character that small repairs are not reported. Despite the reduction of clerical work the efficiency of such a patrol system is easily assessed by the rise or fall of maintenance costs and production losses in each patrol area.

There is a very sound case for patrol or similar systems which ensure that a screw-driver, a spanner, or oilcan are used in the right place at the right time, for surveys of many failures resulting in expensive maintenance have indicated that more than three-quarters of such failures

are initially due to loose screws, nuts, or bolts, or inadequate lubrication.

Nevertheless, it should be realised that adequate records of work performed and results achieved provide management with invaluable information on the effectiveness of the maintenance department's activities. The key to economical maintenance is good organisation based on adequate information. It should not be overlooked that one purpose of records is to provide tangible evidence that preventive maintenance pays and continues to pay.

#### Results of Preventive Maintenance

In the course of a visit I made to the United States last year I had the opportunity of studying general developments in production methods, and became increasingly aware of the tremendous strides the Americans have made in transforming maintenance from an essentially ancillary activity restricted to the repair or overhaul of unserviceable plant into a key function of industry which directly increases productivity and reduces factory costs. Over a period of years irrefutable evidence has been accumulated of the effect of preventive maintenance on plant life, machine availability, and maintenance costs. There was a widespread realisation

that preventive maintenance constituted a relatively simple and effective method of increasing the yield from existing resources of machines and men without the necessity for large investments in capital equipment.

At Cadillac motors the introduction of preventive maintenance had reduced breakdown time by more than 80% over a period of five years. At the start, about three-quarters of the maintenance group was assigned to preventive maintenance and the rest to breakdowns, but by the end of the first year, both the preventive maintenance group and the breakdown group had been reduced, the latter by half. In another firm, the introduction of preventive maintenance reduced press shop maintenance costs by 40%, and press shop requests for emergency repairs eventually decreased from an average of 20 per week to 3. Following the introduction of preventive maintenance in a factory in which 40 motor generator sets, 118 items of switchgear, and 100 cranes and hoists were installed, no generator or switchgear failures occurred in seven years, and only three half-hour delays per year resulted from crane and hoist failures. The life of lathes and grinding machines had been increased by 5-10%, shot blasting machines by 8%, and the life of automatic lathes doubled. The cost of maintaining trucks and tractors was reduced by half.

## High Speed Analytical Control of Light Alloys

A NEW system of metallurgical quality control, introduced into this country for the first time by T.I. Aluminium, Ltd., is said to enable analytical work that would normally take six chemists one day to be carried out by two men in one hour. The system, also applicable to other metal-producing industries, is the most revolutionary advance yet made in rapid and accurate control of metal composition and it is now considered possible to complete an accurate analysis for up to 13 elements in less than 4 minutes.

In the production of high strength and other special aluminium alloys, the alloying elements must be controlled to close limits. Due to the time factor, it has previously been necessary to verify the composition after the metal was cast. If the metal failed to meet specification requirements, it was rejected for remelting, with consequent production hold-up. With the method now in operation, the speed with which results are obtained permits the checking of the metal in the furnace before it is cast, and any corrections necessary can be made immediately.

The new technique is based on a complex apparatus known as the A.R.L. Production Control Quantometer, which is an electronically operated form of direct-reading spectrograph. As one of the essentials in the successful use of the instrument is the maintaining of a constant and delicate optical focus, it is housed in a special laboratory sited in a corner of the foundry, under controlled conditions of temperature and humidity.

The three components of the apparatus are a source unit for exciting an arc, or spark, from the metal to be analysed; a spectrometer for measuring the relative intensities of the light wave-lengths existing in the arc; and a recording console giving direct interpretation of the results.

Test samples, cast at the furnace side, undergo a high



speed machining operation to provide a pencil-shaped or flat plate specimen, which is inserted in the source unit. An arc is struck from the metal surface and the light is optically directed into the spectrometer, where the relative intensities of the component light wave-lengths (or spectrum lines) are measured by photo-multiplier valves.

These valves charge condensers which, in turn, operate through an amplifier to feed the high speed recorder, from which the results are read and signalled to foundry control centre. The exact amounts of up to 13 different elements in one sample are determined simultaneously in less than 4 minutes, including the time taken in preparation of the test piece.

# NEWS AND ANNOUNCEMENTS

## Restricted Use of Non-Ferrous Metals

### Further Measures Agreed by O.E.E.C.

FURTHER steps have been taken by the Council of the Organisation for European Economic Co-operation to restrict the use of certain non-ferrous metals, shortages of which are causing concern to European manufacturers. At the same time the Council has reviewed the success of measures put into effect last year.

In the case of nickel, nickel alloys and nickel plating, the Council decided that member countries should prohibit their use in the manufacture of nearly 500 commodities, ranging from domestic appliances to machinery and motor-vehicles. Member countries will be entitled to grant exemption to those measures in certain cases for their home markets, to allow manufacturers to dispose of stocks, and to maintain trade with non-member countries. The export to member countries of commodities on the list which contain nickel in prohibited quantities is banned. A number of countries have already put these measures into effect.

The Council urged member countries to promote the use of case-hardening and constructional steels containing neither nickel nor molybdenum, but alloyed with manganese, chrome-vanadium, and possible boron. Member countries will in future take all possible steps to prohibit the manufacture or use of case-hardening and constructional steels containing more than a certain percentage of nickel and molybdenum. Exceptions to this prohibition will be allowed under circumstances similar to those permitted in the case of nickel and nickel alloys.

The Council also examined the implementation of last September's decision to prohibit the use of copper and copper alloys in the manufacture of a long list of commodities, and concluded that member countries had carried out their undertakings in a satisfactory manner. Exemptions were permitted in this case also, but the Council agreed that the quantity of copper involved was not sufficient to influence the effect of the prohibition.

In the case of zinc and zinc alloys, the Council took note that since the supply position had improved, it would not be necessary at the moment to introduce a common list of end-use prohibitions. This list has however, been prepared and agreed by member countries and can be applied should the necessity arise. A report on measures for economising in the use of manganese was examined, but the Council took no decision in this instance.

It is the intention of the Organisation to keep these measures constantly under review, so that they may be relaxed should the supply situation improve.

## Coronation Souvenir Manufacture

FIRMS intending to manufacture coronation souvenirs will be interested in the following announcements concerning design standards and metal licences:

*Design Standards.* The Council of Industrial Design has, at the request of the Board of Trade and with the approval of the Home Office, the Lord Chamberlain and the Keeper of the Privy Purse, formed a Coronation Souvenirs Committee with the object of promoting a high

standard of design in souvenirs made by British manufacturers for the Coronation. Submissions are invited from individual firms working in any material and designs may be submitted, in the first instance, in the form of drawings, models or prototypes. Designs that are accepted by the Committee will be included in a new section of the Council of Industrial Design's *Design Review* devoted exclusively to Coronation souvenirs. The Committee will meet twice a month. Lists of approved souvenirs and their makers will be issued at intervals to the press and the retail trade, and an exhibition for retailers will be held in London at a later date. The Secretary of the Committee may be consulted by manufacturers on the conditions that should be observed in designing souvenirs for this Royal occasion. This Coronation Souvenirs Committee is complementary to the Coronation Medal Panel which, as already announced, has been set up by the Royal Mint, the Federation of British Industries and the Council of Industrial Design and will work in close collaboration with the Royal Mint Advisory Committee of which H.R.H. The Duke of Edinburgh is President. All souvenirs should be submitted to the Secretary, Coronation Souvenirs Committee, Tilbury House, Petty France, S.W.1 (Whitehall 6322); all medals should be submitted to Miss S. Milman, Coronation Medal Panel, Goldsmith's Hall, Foster Lane, Cheapside, E.C.2.

*Metal Licences.* The Board of Trade has now decided to license reasonable requirements of copper and zinc alloys for the manufacture of medals and other small coronation souvenirs, not only for export, but also for the home market. The present severe restrictions on the use of nickel and nickel alloys will, however, continue in force. Applications for licences should be made in accordance with the normal procedure to the Regional Offices of the Board. The extent to which licences can be granted will depend upon the availability of the controlled metals. The use of copper and alloys containing substantial quantities of copper should be avoided if any other material (except nickel and nickel alloys) is suitable. The licences will be issued under the Copper and Zinc (Prohibited Uses) (Board of Trade) Orders. A licence will not carry entitlement to any allocation of metal.

## British Machine Tools and Canada

MR. ROBERT W. ASQUITH, President of the Machine Tool Trades Association, who were organisers of the British Machine Tool Section of the Canadian International Trade Fairs of 1950 and 1951, entertained the Rt. Hon. C. D. Howe, Canadian Minister of Trade and Commerce and Minister of Defence Production, at an informal dinner held on Monday, June 2nd, at the Royal York Hotel, Toronto.

In addition to the Minister there were present Mr. F. Bull, Deputy Minister, Trade and Commerce; Mr. R. M. Brophy, Deputy Minister, Defence Production; Mr. J. S. P. Armstrong, Agent-General for Ontario in London; Mr. Glen Bannerman, Director of Canadian Government Exhibition Commission; Col. F. J. Lyle, O.B.E., Director Trade and Industry Branch, Department of Planning and Development for the Province of Ontario; Mr. J. Rudel, President Canadian Machine

Tool Dealers' Association ; Mr. Keith Jopson, C.M.G., O.B.E., Senior Trade Commissioner for the United Kingdom ; Mr. J. Balcon, U.K. Trade Commissioner, Toronto ; Mr. C. Empson, C.M.G., H.M. Minister (Commercial), Washington, U.S.A. ; Mr. Ed. Barker, President Modern Tool Works, Ltd. ; Mr. M. Bowman, Director Modern Tool Works, Ltd. ; Mr. A. C. Wickman, President A. C. Wickman (Canada), Ltd. ; Mr. H. Style, President, John Inglis Co. ; Mr. Horace T. Hunter, President, Maclean-Hunter Publishing Co., Ltd. ; Mr. B. C. Gardner, President, Bank of Montreal ; Sir Holland Goddard, Member of the Executive Committee, Machine Tool Trades Association ; Mr. S. Marsland, Machine Tool Controller, Department of Defence Production.

The occasion was a completely informal get-together, but it is understood that in the course of talk over the dinner table assurances of the continued interest of the British Machine Tool Industry in the Canadian market were given and matters of mutual interest concerning that trade were freely discussed.

Nothing has been more striking than the increase in recent years of the orders placed for British machine tools for Canada. In 1949 the value of orders received from Canada for metalworking machine tools amounted to £467,141 and for woodworking machines, £29,091, giving a total of £496,232. The corresponding figures for the year 1951 had risen to £4,243,139 for orders for metalworking machine tools and £261,293 for woodworking machines, giving a total of £4,504,332.

### Magnesium Advisory Committee

THE Magnesium Advisory Committee announces that it has appointed Wenham Brothers & Co., of 21, Bennett's Hill, Birmingham, 2 (Tel. No. : Midland 0994/7) as Permanent Secretaries. This Company also has an office in London at 15, Tooks Court, E.C.4 (Tel. No. : Chancery 7161), but communications should normally be addressed to Mr. A. M. M. Burdon-Cooper at the Birmingham office.

### Plant Engineers Fuel Policy Committee

At the national conference held at Harrogate last month, unanimous consent was given to a proposal that the Institution of Incorporated Plant Engineers should set up its own National Fuel Policy Committee. The proposal was made by Mr. L. G. Northcroft, O.B.E., Chairman of the Education Committee of Incorporated Plant Engineers. In the matter of fuel policy, said Mr. Northcroft, there was a point where operational efficiency "comes to a dead end, due to some outside influence" Outside the efforts of the individual fuel user to establish a higher all-round standard of industrial fuel efficiency, there had seemed to be "a mass of passive obstruction" in the way of a properly co-ordinated policy for fuel utilisation.

The views of the users of fuel and power were heard far less than the views of the suppliers of fuel and power. The Institution of Incorporated Plant Engineers, representing the technical users of fuel and power in industrial production and services, had the competence to offer views and recommendations on fuel policy to the Government and to any advisory bodies set up by the Government. That could be done, to the greater

benefit and welfare of the community, through a National Fuel Policy Committee of the Institution of Incorporated Plant Engineers, whose members are the key administrators and executives responsible for keeping the wheels of industry turning.

### Personal News

MR. H. G. HERRINGTON has been appointed Managing Director of High Duty Alloys, Ltd.

MR. J. A. MONKS has been appointed Managing Director of Electric Furnace Co. Ltd., jointly with Messrs. D. F. CAMPBELL and W. S. GIFFORD.

MR. W. J. MILLER has been appointed Managing Director of the EFCO Engineering Co. Ltd., Burton-on-Trent.

MR. W. SYMES has resigned from the Board of Metropolitan-Vickers Electrical Co. Ltd., and from the position of Works Manager, Trafford Park Works, in order to take up the appointment of Manufacturing Consultant, Associated Electrical Industries, Ltd. He will be available to all the A.E.I. Group Companies to advise on general manufacturing problems. MR. A. C. MAIN, Assistant Works Manager at Trafford Park, has been appointed Works Manager in succession to Mr. Symes and has been elected a Director of the Metropolitan-Vickers Co.

MR. E. MEAD has been appointed Assistant Manager of the London Office of Metropolitan-Vickers Electrical Co. Ltd. ; MR. R. ALLEN, Assistant Manager of the Birmingham Office ; and MR. T. GILL, Manager of the Swansea Sub-Office.

MR. N. MCPHERSON has been appointed a Director of Aluminium Corporation, Ltd.

MR. F. H. BROOKS, General Works Manager of The Wellman Smith Owen Engineering Corporation, Ltd., has been appointed a Director of the Company.

MR. H. M. H. FOX has been appointed a Director of Smith-Clayton Forge Ltd. Formerly General Manager, Mr. Fox has been with the Company for the past 19 years. PROFESSOR E. N. DA C. ANDRADE, F.R.S., has resigned from the offices of Director of the Royal Institution, Resident Professor in the Institution, Fullerian Professor of Chemistry, Superintendent of the House and Director of the Davy-Faraday Laboratory.

MR. H. W. HUNT, Chairman of Midland Heat Treatments, Ltd., has resigned the Managing Directorship. MR. E. G. LEES succeeds him and MR. C. E. ELWELL becomes a Director.

MR. S. C. TYRELL, Local Director and Chief Accountant of Newton Chambers and Co. Ltd., has been elected President of the Institute of Cost and Works Accountants.

### Obituary

WE regret to record the deaths of the following :—  
MR. THOMAS TROTT, late Chairman and Managing Director of Thomas Andrews and Co. Ltd., who passed away at his residence at Southbourne on Saturday, May 10th. Mr. Trott had been associated with the Company for 50 years.

MR. H. ROBINSON, Senior Partner of H. Robinson, Ltd., of Montevideo, agents in Uruguay for Foundry Services, Ltd.

# RECENT DEVELOPMENTS

## MATERIALS : PROCESSES : EQUIPMENT

### New Grade of Cemented Carbide

The range of Cutanit cemented carbides has recently been extended by the introduction of a new grade "W." This grade contains no tungsten carbide, but has chromium carbide as its principal constituent. It has high resistance to chemical corrosion, particularly by acids, and in addition has good wear resistance. It has a specific gravity almost half that of the normal tungsten carbide grades and has a coefficient of thermal expansion very similar to that of steel. This latter property gives it a great advantage over the tungsten carbide grades for applications in which the carbide is fixed into a steel matrix either by shrinking or brazing, especially where it is subjected to heat during working.

Grade W is recommended specially for parts subjected to wear and/or chemical corrosion, and, unlike other cemented carbides in the Cutanit range, is not intended for use as cutting tool tips. Development work is now just completed and a limited number of trial pieces can be supplied through the Cutanit distributors, Messrs. William Jessop & Sons, Ltd., Sheffield.

*Metro-Cutanit, Ltd., 160, Piccadilly, London, W.1.*

### Carbon Dioxide Indicator

THE fuel position to-day is such that it is essential that as much effective heat as possible be obtained from each unit of fuel burnt. The control of combustion air is one of the significant factors in achieving fuel economy, for, no matter what fuel is used, or how it is consumed, one of the biggest losses in converting the fuel into useful energy is caused by excess air in the chimney. To cut this loss to a minimum it is necessary to take periodic measurements of the CO<sub>2</sub> in the flue gases so that any necessary adjustments may be made to the air supply. It is not possible to gauge furnace-firing efficiency by observing the fire, or by looking at the top of the chimney, as an apparently perfect chimney may still have a poor CO<sub>2</sub> reading; nor is the density of any visible smoke a correct indication of the losses due to unburnt gases.

A simple and handy instrument called the Fyrite has recently been introduced into this country by the distributors, Shandon Scientific Company, with the object of making CO<sub>2</sub> testing a practical proposition for all furnace users. Instead of CO<sub>2</sub> testing being a lengthy and expert business, it is possible for non-technical personnel to obtain, in 40 seconds, a CO<sub>2</sub> reading with an accuracy better than  $\frac{1}{2}\%$ .

The instrument, which is supplied in a strong metal-carrying case complete with gas sampling accessories, is dumbbell shaped with a spring-loaded gas-inlet valve at the top, a reservoir of CO<sub>2</sub>-absorbing fluid at the base, and a scaled tube between. In operation, the sampling tube at one end of the rubber hose (which has in its length a filter and a rubber bulb) is inserted into the flue pipe, and the connector plug at the other end of the hose is pressed down on the spring-loaded valve, thus opening a passage into the Fyrite. The gas sample is then pumped into the Fyrite by about 18 squeezes of the rubber bulb and automatically locked in the instru-

ment by lifting the finger from the connector plug at the last bulb stroke. The Fyrite is then turned upside-down and back again to mix the gas sample with the absorbing fluid. Absorption of the CO<sub>2</sub> creates a partial vacuum in the instrument so that the absorbing fluid is sucked up into the centre tube in an amount equal to the CO<sub>2</sub> absorbed. The scale reading at the top of the fluid column is the percentage of CO<sub>2</sub> in the flue gas sample tested. The reading is not affected by the presence of any other gas in the sample.

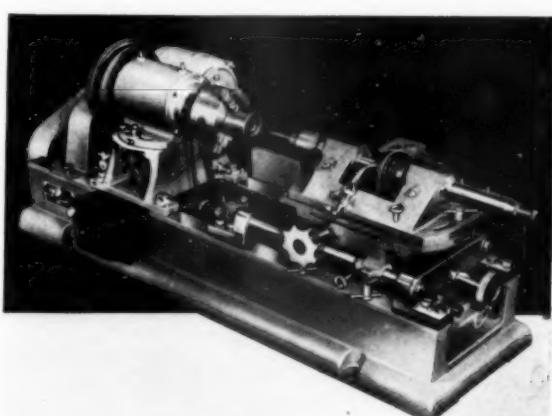
*Shandon Scientific Company, 6, Cromwell Place, South Kensington, S.W.7.*



### Die Lapping and Polishing Machine

Recent improvements in the design of their type 2015/1 high-speed universal lapping and polishing machine introduced by the makers, Rudkin & Riley, Ltd., enable every possible operation on wire, rod and tube drawing dies to be carried out fully automatically on this machine. The operations for which the 2015/1 is intended include the lapping and polishing of angles, bores and radii of steel, tungsten carbide and other hard-metal dies.

The headstock, carrying a 4-in. self-centring chuck, is power-driven by an arrangement which imparts a smooth radial movement; this action enables the radius



in round dies to be polished automatically at high speed. Ample adjustment is provided to govern the movement required, which may be measured from a graduated scale conveniently mounted for easy reading. Radial movement is controlled by a clutch with a start-stop device situated in front of the machine whereby the radial movement may be stopped instantly. Accurate slots in the headstock allow the head to be set at any desired angle, or parallel for bore sizing and polishing. Quick-action lock screws secure the headstock after setting.

Exact positioning of the lap is possible by means of the lap carriage longitudinal micro adjustment. With this device the operator moves the lap carriage into the approximate working position, then by push-pull action through a spring-loaded split nut final accurate setting is obtained. The lap carriage is set to correct height and is adjustable transversally in both directions: the whole carriage may be moved 6 in. for adjustment along two direction bars. The upper portion of the lap carriage has been designed to swivel to the degrees required by registering the indicating line with an engraved scale—thus the lap engages with the die at the required angle. A locking screw registers the swivel lap carriage in the zero degrees position, whilst a dowel registers and retains the lap in a true axial line for bore sizing and polishing.

The standard 2015/1 is fitted with a 4-in. self-centring chuck but larger chucks, or stepped collets, will be fitted where required. As an alternative to bench-mounting, a cabinet stand will be supplied with the machine where desired.

As an example of the performance of the machine, the makers state that the bore of a tungsten carbide die was opened out from 1.250-1.275 in., and the mirror finished restored, in a total time of 45 minutes.

*Rudkin & Riley, Ltd., Aylestone, Leicester.*

### Hand Lever Shears

A RECENT addition to the Besco range of metal working machinery is the new hand lever shear, made in two sizes. The frame is a forging from  $\frac{5}{8}$  in. steel section of adequate strength for top capacity working, while the cutting mechanism is actuated by a 30 in. hand lever and toggles constructed of steel plate. The BS1P model has 4½-in. blades and is capable of cutting plate up to  $\frac{1}{8}$  in., rounds up to  $\frac{3}{8}$  in. and flats up to  $\frac{1}{16}$  in.  $\times$  2 in., whilst

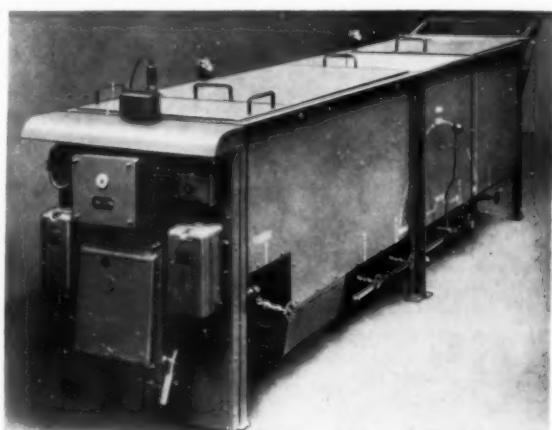


the corresponding limits for the 6-in. blades of the larger model, BS2P, are:  $\frac{5}{8}$  in. plate;  $\frac{7}{16}$  in. rounds; and  $\frac{3}{16}$  in.  $\times$  2 in. flats. The cutting blades are easily removed for servicing or renewal.

*F. J. Edwards, Ltd., 359-361, Euston Road, London, N.W.1.*

### Alkali Degreasing Plant

THE latest addition to the well-known range of industrial degreasers made by the Orcene Co., Ltd., is the Orco Vat alkali degreasing plant which will be of interest to a wide variety of industries. The principle of alkali degreasing is not new, and has been tried and proved over many years, but this new Orco Vat equipment has been designed to give more efficient results from the process than have been obtainable hitherto.



Alkali degreasing is recommended when a chemically clean surface is required. This high degree of cleanliness is particularly necessary prior to such processes as vitreous enamelling, electroplating, tin printing and other types of decorative finishing where complete freedom from oils and chemicals is essential. It is possible, also, by adjustment to the alkali solution, to etch aluminium surfaces during the degreasing, either for decorative purposes or to provide a "key" for subsequent finishing processes.

The Orco Vat is believed to be the only equipment of its kind with a self-contained oil separator. This motor-driven separator is positive in action and prevents the absorption of oil into the alkali solution. It also keeps the surface of the alkali free from oil so that work-pieces are not contaminated as they are withdrawn, and so efficient is this auxiliary equipment that the separated oil can be reclaimed for subsequent use. The plant is supplied complete with all electrical switchgear and immersion heaters.

The manufacturers claim that the cost of chemicals for the process is only two-fifths of that for emulsion-type degreasers, although it may be found that heating costs are slightly higher. This alkali degreasing plant can be operated by unskilled labour: even the strength of the alkali is automatically indicated by a system of coloured lights which show when it is necessary to regenerate the solution.

*The Orcene Co., Ltd., Warwick.*

# CURRENT LITERATURE

## Book Notices

### WIRE ROPES IN MINES

Proceedings of the Conference held at Ashorne Hill, Leamington Spa, in September, 1950. 828 pp., demy 8vo, illustrated, bound in cloth. The Institution of Mining and Metallurgy, Salisbury House, London, E.C.2. 50s. or £7 postage included.

THIS volume contains the text of 18 papers presented to the Conference on Wire Ropes in Mines, which was held at Ashorne Hill in September 1950, together with a report of the subsequent discussion and a summary of conclusions and recommendations. An indication of the field covered by the Conference may be obtained from the titles of the 18 papers: "Manufacture and Properties of Rope Wire," by R. S. Brown; "Steel Wire Ropes used in Mining Practice," by the Wire Rope Manufacturers of the United Kingdom; "Wire Rope Practice in British Coal Mines," by B. L. Metcalf; "Wire Rope Practice in Ontario Mines," by the Ontario Mining Association Committee on Hoisting Ropes; "Winding Rope Practice on the Witwatersrand," by J. Dolan, W. G. Jackson and L. T. Campbell Pitt; "Some Aspects of American Wire Rope Practice in Mining," by Harlan A. Walker and Josiah Gerrans; "Deep Winding on the Kolar Gold Field," by J. W. P. Chalmers and A. H. P. FitzPatrick; "Studies on the Testing of Wires for Hoisting Ropes," by Paul Teissier; "Belgian Rope Practice," by Y. Verwilt; "Koepf Winding Ropes for Main Shafts in the Netherlands," by J. W. Kleimbentink; "Winding ropes in the Ruhr," by H. Herbst; "Dynamic Stresses in Wire Ropes for use on Vertical Hoists," by P. J. Pollock and G. W. Alexander; "Failures in Wire Ropes in British Colliery Practice," by A. E. McClelland; "Wire Rope Research at the Ontario Research Foundation," by I. A. Usher and L. W. Sproule; "Winding Rope Testing Practice in Metalliferous Mines, Queensland, Australia," by I. W. Morley; "A Survey of Government Regulations, with Particular Reference to Safety Factors," by M. A. Hogan; "Multi-layer Coiling of Locked Coil Ropes as used on the Skip Haulage at Broken Hill South, Ltd.," by J. C. Crace and E. Goninan; and "Electronic Inspection of Mine Ropes," by William Simpson. The volume is fully indexed.

### THE GAS WELDING OF ALUMINIUM

The Aluminium Development Association, Information Bulletin No. 5. 68 pp., 41 illustrations, paper cover. 2s.

THIS publication is a major revision of the former Bulletin No. 5 which was entitled "Fusion Welding of Wrought Aluminium Alloys." In preparing this revision, information dealing with metal arc welding has been removed but will be included with comparable information in a new Bulletin No. 19 on "The Arc Welding of Aluminium." Bulletin No. 6 continues to deal with resistance welding.

The present book begins with an outline of the fusion welding methods and then deals with those modifications to procedure that result from the characteristics of aluminium. It is of course understood that the problems presented by the work necessarily differ as does the

variation in skill between the welders. Nevertheless experience very quickly enables welders trained in other metals to become proficient with aluminium.

The description of gas welding processes is divided as between; fuel gases; flame types; equipment; fluxes, and filler metal. Welding procedure is then described beginning with the preparation of parent metal, dealing with techniques for horizontal and vertical welding and ending with an account of after-welding treatment. There are shorter notes on atomic hydrogen welding and other processes.

The notes on inspection of welds, the properties of gas welds and data for estimating costs conclude the main portion of the book, and are followed by appendices giving the specified composition of aluminium welding wire (B.S.1474) and a table of aircraft specification numbers in relation to B.S.1470-1477 and 1490.

### A BIBLIOGRAPHICAL SURVEY OF GERMAN AND JAPANESE EXPERIENCE IN THE EXTRACTION OF LOW-GRADE NON-FERROUS ORES AND THE RECOVERY OF NON-FERROUS METALS AND COMPOUNDS

Report R.M.S.-3 of the Technical Information and Documents Unit of D.S.I.R. 100 pp., paper cover. Published by H.M. Stationery Office, London. 3s. 6d. (90 cents U.S.A.) by post 3s. 8d.

THIS publication is a bibliographical survey of information on the recovery of non-ferrous metals from waste materials and on their extraction from low grade ores. The survey is based on German and Japanese experience in this work before and during the war and includes a list of more recent U.S. reports held by T.I.D.U. The Report deals with mining and ore dressing and with the smelting and recovery of base metals. It also covers the production of and recovery of non-ferrous metals. Sections dealing with precious metals, rare earth metals and selenium are included. The waste materials from which metals have been recovered include slags, scrap metals, waste liquors, flue dusts and spent catalysts. Grinding dust, swarf and even floor sweepings from engineering works can be treated for the recovery of scarce metals.

### UNITS AND STANDARDS OF MEASUREMENTS.—III

Published for D.S.I.R. by H.M. Stationery Office and obtainable only from the National Physical Laboratory, Teddington, Middlesex. 9d. (25 cents U.S.A.) by post, 10d.

"ELECTRICITY," the third book let in the series, "Units and Standards of Measurement employed at the National Physical Laboratory" deals with current, voltage, resistance, power, energy, inductance, capacitance, frequency, etc., and gives an account of the history of the subject and of international co-operation to ensure uniformity. It defines the units employed at the N.P.L. for the measurement of electrical quantities and the standards by means of which these units are determined and preserved. They are made applicable to present day practice by reference and working standards which can be calibrated to an accuracy of about one part per

million; details of these are included. There is also a section on radio frequency standards. A daily programme of standard frequency transmissions from station MSF at Rugby is monitored by reference standards maintained at the N.P.L.

## Trade Publications

"THE Diamond Tool Industry in 1951" summarises articles and patents published during 1951 and having a special bearing on the development of industrial diamonds. Copies are available free of charge, from the Industrial Diamond Information Bureau, 32-34, Holborn Viaduct, London, E.C.1.

THE latest Vitreosil Tubing and Rod List issued by The Thermal Syndicate, Ltd., Wallsend, contains the February, 1952, prices for tubing made in the four grades of Vitreosil: sand surface, glazed, satin surface and transparent. Among typical applications for tubes are gas sampling tubes, electric furnace construction, and pyrometer insulators and sheaths, whilst the rods and bars have been found useful in various branches of research.

A LEAFLET issued by Richd. W. Carr & Co., Ltd., Sheffield, makers of tool steels and small tools, gives particulars of the service offered by their Birmingham Heat Treatment Dept., at Bolton St., Bordesley, Birmingham, 9. The modern equipment includes salt baths, controlled atmosphere furnaces, thermostatically controlled air-tempering furnaces, etc., suitable for a wide range of heat-treatment operations, including case-hardening, salt nitriding, isothermal treatment, normalising, annealing and hardening and tempering.

THE first issue of a journal with the title of *Instrument Engineer* recently made its appearance. Published by George Kent, Ltd., Luton, it is intended for the use of the instrument engineer who is a comparatively newcomer to industry, and it may be some considerable time before his status and the qualifications he requires are clearly defined. In view of the wide range of interests to be met, there will be articles making available the latest information concerning recent advances in instrument technique, and it is hoped to devote considerable space to applicational engineering. In this issue are articles on "Oxygen Analysis," "Special-Purpose Applications of 'On-Off' Control (Heat Treatment Furnaces)," "The Application of Automatic Control to a Gas Producer," and "The History of Flow Measurement by Differential Pressure. I—The Work of Venturi." The price of the journal is 2s. 6d. per copy with a subscription of 5s. for the two issues published each year.

BANDSAW machines for foundry and general purposes are featured in the latest "Midsaw" brochure issued by The Midland Saw and Tool Co. Ltd. They include a 22-in. fixed table single-speed machine; a 30-in. moving table 8-speed machine for general and heavy-duty non-ferrous work; two 36 in. models, one fixed table 8-speed, and the other moving table auto-feed and return, for large heavy castings and special heavy duty work. Primarily designed for the cutting of aluminium or magnesium castings of the flat type is the horizontal bandsaw, and for cutting stainless steel and Nimonic castings there is a vertical friction-cutting bandsaw.

WE have received from the General Electric Co. Ltd. a publication dealing with G.E.C. horizontal batch type electric furnaces. As can be seen from the illustrations, these cover a range of sizes and ratings for operation at temperatures up to 1,050° C. Furnaces can be built to meet special requirements and the Company specialises in the supply of complete installations including handling machinery, controlled atmosphere plant and temperature control equipment.

WE have received from Fossain-Wycliffe Foundries, Ltd., the latest editions of brochures concerning two of their products. C.Y. Alloy was first placed on the market in 1934 after many years of research in an attempt to produce a material which would resist abrasive wear, and thus reduce the cost of frequent replacement of parts and, above all, maintenance labour. The success which greeted these efforts is shown by the fact that the alloy, as can be seen from the illustrations of typical parts, is being used in a wide range of industries including collieries, brick, pipe and tile works, quarries, sand and gravel pits, cement works, mines of all types, steel works, etc. The Fossain Penetral process is used to enhance the resistance to scaling or oxidation of mild steel, thus enabling cheaper and more easily fabricated articles to be used in high temperature applications in place of heat-resisting alloys containing nickel and chromium. Details are given of the physical properties of treated articles and of a number of industrial applications.

THE improvements effected in cast iron by additions of zirconium in various proportions have been known and practised for some time. As a ladle addition, in the form of a silicon-manganese-zirconium alloy, it has been used to produce structural improvement in high-strength cast irons. It is only recently, however, that it has been possible to attain this advantage from a cupola addition, having the alloy available in a convenient form. "Bemeo" Publication No. 151 gives details of the zirconium-silicon briquettes made for this purpose by British Electro Metallurgical Co. Ltd., Wincobank, Sheffield. Zirconium when added in this manner is a pronounced sulphide former, can replace manganese additions, promotes the formation of a ferritic matrix, reduces hardness, refines the graphite structure and improves machinability.

## Books Received

"Ferromagnetic Properties of Metals and Alloys," by K. H. Heselitz. Eleventh volume in the series "Monographs on the Physics and Chemistry of Materials." 317 pp. inc. appendices and indices. Oxford, 1952. Geoffrey Cumberlege. 40s. net.

"Tungsten" (A treatise on its metallurgy, properties and applications). By Colin J. Smithells, M.C., D.Sc., F.I.M. Third Edition. 326 pp. inc. 225 illustrations. London, 1952. Chapman and Hall Ltd. 75s. net.

"Manufacturing Processes," by Myron L. Begeman. Third Edition. 608 pp. inc. numerous illustrations. New York and London, 1952. John Wiley and Sons, Inc., and Chapman and Hall Ltd. \$6 or 48s. net.

"Antimony—Its Geology, Metallurgy, Industrial Uses and Economics," by C. Y. Wang, M.I.M.M. Third edition revised. 170 pp. inc. appendix and index. London, 1952. Charles Griffin and Co. Ltd. 25s. net.

# METALLURGICAL DIGEST

## Copper-Clad Aluminium Can Conserve Copper in Many Uses

By John L. Everhart

DEVELOPED originally for service in the electrical industry, copper-clad aluminium is finding new fields of application where copper is scarce or where properties not available in a single metal are needed.

Copper-clad aluminium is manufactured with copper on one or both sides. The most popular applications for single clad material call for an 80/20 ratio—80% aluminium, 20% copper by thickness—but other ratios can be supplied with the copper running from 5 to 50% of the total thickness. In double clad, the most common ratios are 5/90/5 and 10/80/10. The minimum amount of copper for a given overall thickness can best be determined by studying the application. Where an impervious layer of copper free from pinholes is required, a minimum thickness of copper of 0.001 in. is recommended. Where the copper layer is employed merely for ease in soldering, and an occasional pinhole is not objectionable, thinner layers can be used. At present, copper-clad aluminium is available commercially in the form of sheet or strip and in the form of bus-bar. Tubing has been announced also, while wire has been produced in limited quantities.

Copper can be bonded to aluminium by a combined welding and hot-rolling process, but the production of a satisfactory composite metal requires careful control. A compound layer may form at the interface under improper bonding conditions or subsequent annealing. As this compound is quite brittle, the working properties of the metal can be seriously affected. This makes it essential that the manufacturing technique should include all precautions necessary to avoid the presence of this compound. As no binding agent is employed, the properties of the clad metal produced are a combination of those of copper and aluminium.

The physical and mechanical properties of the usual commercial grades are reproduced in separate tables. The

thermal and electrical conductivities of copper-clad aluminium depend upon the ratio of copper to aluminium but in general are the same as the theoretical values for these ratios, the only exception being that there is no additional thermal resistance at the bond such as would result if separate sheets of the metal were merely held together by pressure. In, general, the mechanical properties resemble those of aluminium more closely than those of copper.

The copper and aluminium surfaces of the composite sheet react with chemicals in the same manner as electrolytic copper and commercial aluminium. Parallel tests made by exposing copper-aluminium-copper sheets and electrolytic copper to high-sulphur flue gases, ammonia, steam and

water showed similar corrosion properties for both materials. The possibilities of corrosion-couples or galvanic action should not be overlooked, however, and for use in some environments it would be necessary to protect the cut edges of the sheet to prevent such effects.

For fabricating the material it is recommended that annealed sheet be used for spinning, deep-drawing, sharp bending and embossing. Half-hard sheet can be employed for shallow shaping while hard sheet is suitable only for punching and moderate bending. Recommended reductions for deep-drawing are 40 to 43% for the first draw; 20 to 25% for the second; 15 to 18% for the third; and 14 to 15% for the fourth. In spinning, the composite sheet should be upset gradually, following the practice employed with copper and brass rather than that used for aluminium. The material can be machined satisfactorily with similar tools as are used for aluminium. As a general rule, in drilling, cutting threads, or similar operations, it is preferable to place the

PHYSICAL PROPERTIES OF COPPER-CLAD ALUMINIUM

Clad Material			Density, Lb./Cu. In.	Specific Gravity	Thermal Cond., Btu/Hr./Sq. Ft./F°/F	Coef. of Exp., per °F 32-212 F	Elect. Res., Microhm-Cm	Elect. Cond., % IACS	Mod. of Elast., Psi
Cu	Al	Cu							
10	90	...	0.12	3.33	137	13.0	2.73	63	10,400,000
5	90	5	0.12	3.33	137	13.0	2.72	63	10,100,000
20	80	...	0.14	4.00	144	12.5	2.63	66	10,800,000
10	80	10	0.14	4.00	144	12.5	2.59	67	10,800,000
30	70	...	0.16	4.50	154	12.0	2.52	69	11,000,000
15	70	15	0.16	4.50	154	12.0	2.44	71	10,500,000
40	60	...	0.19	5.22	...	...	2.36	73	...
20	60	20	0.19	5.22	...	...	2.28	76	...
50	50	...	0.21	5.85	...	...	2.20	78	...
25	50	25	0.21	5.85	...	...	2.17	79	...

MECHANICAL PROPERTIES OF COPPER-CLAD ALUMINIUM

Clad Material	Condition	Yld. Str., 0.2% Offset, Psi	Ten. Str., Psi	Elong. %	Vickers Hardness No. 100 G. Load 0.020-In. Sheet		Bend No. 5 T	Erichsen Value Mm (0.020-In. Sheet)	
					Al-Side	Cu-Side		Cu-Outside	Al-Outside
10 90	Annealed Cold-rolled*	5,700-7,100 18,500-21,400	12,800-15,600 25,600-28,500	28-31 5-7	33	70	14-22	9-2	9-1
5 90	5 Annealed Cold-rolled*	5,700-7,100 19,000-22,800	12,800-15,600 25,600-28,500	28-31 5-7	...	72	8-16	9-0	...
20 90	Annealed Cold-rolled*	6,400-7,800 22,800-25,600	14,200-17,100 27,000-30,900	30-33 5-7	33	70	18-26	9-5	9-4
10 80	10 Annealed Cold-rolled*	6,400-7,800 22,800-25,600	14,200-17,100 27,000-30,900	30-33 5-7	...	70	12-18	9-2	...
30 70	...	7,100-8,000 24,200-27,000	17,100-19,900 28,500-32,800	34-36 6-8	33	69	23-30	9-8	9-7
15 70	15 Annealed Cold-rolled*	7,100-8,000 25,600-28,500	17,100-19,900 28,500-32,800	34-36 6-8	...	70	16-20	9-45	...

\* Cold-rolled, 50% reduction in thickness.

copper side on top in working material clad only on one side. Before cutting threads it is advisable to countersink the pre-drilled hole.

When annealing of the material is necessary it should be done within the range 700 to 750° F., preferably in an accurately controlled furnace or a salt bath. Annealing time should be as short as possible. Exposure at higher temperatures or prolonged exposure below 750° F. results in the formation of a compound layer at the interface which can modify the properties of the

clad material drastically.

Welding requires considerable care as a brittle copper-aluminium compound can be formed on melting. Satisfactory joints can be made by the spot-welding of single clad material if the aluminium surfaces are placed in contact with each other. Soft soldering of the copper layer offers no problem, but hard soldering to the copper layer involves a special technique which allows the aluminium to be cooled artificially to prevent it reaching the melting point.

## Induction Heating for Continuous Heat Treatment of Aluminium Sheet and Strip

By F. C. Hull and H. Scott

WORK on the continuous annealing and the continuous solution heat treatment of aluminium strip is described. Preliminary tests on the relation between the hardness of the product and the temperatures and times of immersion were carried out using a salt bath, leading up to the use of induction heating.

In the preliminary experiments, specimens of cold-rolled 0.020 in. Alclad 24S strip were heated in a salt bath for various times, quenched and aged for 24 hours at room temperature before testing. Complete softening required 20 secs. at 650° F. and 5 secs. at 750° F. (Above 700° F. annealing time must be limited, as a slight increase in hardness occurs if held longer than 10 secs.).

Continuous solution treatment of aluminium alloys in preparation for age hardening has even more appeal than annealing for softening or recrystallisation, because of the possibility of reducing the distortion encountered in batch quenching operations. To evaluate the time factor involved, 0.040 in. bare 24S sheet, as cold rolled, was heated in a salt bath, water quenched and aged 24 hours at room temperature. Maximum hardness was obtained in 3 min. at 930° F. Higher temperatures incur danger of grain boundary melting; lower temperatures result in inadequate hardening.

High-frequency induction heating with longitudinal flux is ineffective with non-ferromagnetic strip materials, but R. M. Baker, of Westinghouse

Research Laboratories, overcame this difficulty by developing transverse flux induction heating, wherein the magnetic flux is directed perpendicularly to the surface and through the strip. A 300 kW experimental unit for 54 in. strip is illustrated, and a 1,875 kVA unit was incorporated in a unit built by the United Engineering and Foundry Co. for the Société Centrale des Alliages Legers. With the 54 in. unit aluminium sheet could be heated rapidly from a 60-cycle power supply at an efficiency of 80% or better. When cold-rolled Alclad 24S strip 0.040 × 54 in. was annealed, there was an edge effect, but by blowing air against the edges a nearly uniform hardness was attained across the strip at a throughput of 30 ft. per hour. For production, an attempt was made to avoid edge effect and obtain uniform heating by moving the poles of the inductors laterally to one another, so that the flux through the edges was the same as at the centre. This also enabled adjustment to the actual strip width. The strip was also found to pull over against the extended pole pieces due to electrical currents induced near the edges, so U-shaped copper shields were attached to the edge of each pole structure. Temperature was checked at a point intermediate between the sides and the centre, by a thermocouple placed at the end of a long lever. These modifications and improvements were incorporated by Westinghouse in the 1,875 kVA transverse flux inductor coil for the equipment built for the Société Centrale des Alliages Legers.

High-frequency induction heating with longitudinal flux is ineffective with non-ferromagnetic strip materials, but R. M. Baker, of Westinghouse

## Gallium

By M. Beja

THIS article is an extensive survey covering the history and occurrence of gallium, the extraction of gallium oxide from ores, various industrial by-products, etc., the production and properties of metallic gallium, its alloys and applications, and production figures and prices.

In the section on the extraction of gallium oxide, special reference is made to the recovery of gallium oxide from bauxite in conjunction with the Bayer process. Bauxite contains about 0.007 part  $\text{Ga}_2\text{O}_3$  per 100 parts  $\text{Al}_2\text{O}_3$ . The gallium oxide is dissolved by the caustic soda and accumulates in the recirculated caustic liquor finally reaching an equilibrium concentration of 0.14 parts  $\text{Ga}_2\text{O}_3$  per 100 parts  $\text{Al}_2\text{O}_3$  at French alumina works; 0.30 parts  $\text{Ga}_2\text{O}_3$  per 100 parts  $\text{Al}_2\text{O}_3$  have been reported in the recirculated liquor in American works. Beyond this concentration further gallium introduced with the bauxite precipitates out with the alumina and ultimately finds its way into the aluminium, commercial aluminium containing 0.01–0.02% gallium. The gallium is retained in the anode alloy in the three-layer process of refining aluminium, and can be recovered from the spent anode alloy.

Recovery of the gallium oxide from the equilibrium recirculated Bayer caustic liquor can be effected by differential carbonation, which must be carried out slowly, the alumina tending to precipitate out first, or by precipitating out the alumina by adding lime. Alumina rich in gallium oxide is recovered, from which the gallium is separated by solution in hydrochloric acid and extraction of the gallium chloride with ether, or by solution in caustic soda and electrolysis to deposit the metallic gallium.

Recovery of the gallium oxide from the caustic liquor involves conversion of the latter into sodium carbonate and it is consequently expensive. Attempts made to recover gallium oxide from the liquor without appreciably affecting the latter, or similarly from Bayer alumina, have not, so far, proved successful. A process recently announced for the extraction of the gallium contained in sodium aluminate or alumina makes use of hydrofluoric acid, recovering cryolite and aluminium fluoride as useful by-products.

# LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC  
INSTRUMENTS AND MATERIALS

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## Rapid Photometric Determination of Cobalt in Aluminium Alloys Using Nitroso-R-Salt

By William Stross, M.D., F.R.I.C., and Gertrude Stross, M.D.

*It was found that the method of Haywood and Wood for determining cobalt in steel can be applied to aluminium alloys with only minor adjustments. An even simpler procedure is described here which also has the advantage of enabling the determination of cobalt to be combined with that of copper, nickel and iron from aliquots of the same metal solution. Interference by other metals and nitrite are also discussed.*

**A**MONG the variety of reagents<sup>1</sup> proposed for the photometric determination of cobalt, nitroso-R-salt seems to be favoured by the majority of authors, but it does not yet seem to have been applied to aluminium alloys. As far as the authors know, the only method published for determining cobalt in aluminium alloys is the gravimetric one described in the book of the Aluminum Company of America.<sup>2</sup> It was, therefore, decided to check whether the procedure published for steel by Haywood and Wood<sup>3</sup> could be applied to aluminium alloys without major modifications. The procedure chosen at first was the following.

### Earlier Method

#### SOLUTIONS REQUIRED

These solutions are almost identical with those used by Haywood and Wood.

(1) *Acid Mixture.* With 15% (v./v.) of sulphuric acid (S.G.1.84), 15% (v./v.) of syrupy phosphoric acid (S.G.1.75) and 10% (v./v.) of nitric acid (S.G.1.42).

This is "Spekker Acid" reinforced with nitric acid.

(2) *Sodium Acetate Solution.* 50% (w./v.) aqueous solution of  $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$ .

(3) *Standard Cobalt Solutions*

(a) *Stock Solution.* Dissolve 3.088 g.  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , or 2.981 g.  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$  (A.R. grade) in water and dilute to 250 ml. 1 ml. contains 2.5 mg. of cobalt. Check the cobalt content, e.g. by the volumetric pyridine thiocyanate method<sup>4</sup> and adjust if necessary.

(b) *Calibration Solution.* Freshly dilute the stock solution of cobalt 50 times, so that 1 ml. contains 50  $\mu\text{g}$ . of cobalt.

(4) *Nitroso-R-Salt Solution.* 0.2% aqueous solution.

### Tentative Procedure

Attack a 200 mg. sample in a 250 ml. beaker with 20 ml. of the acid mixture, cover with a watch glass and heat gently to dissolve. Clear any undissolved silicon by adding hydrofluoric acid, 2-5 drops at a time. A total of 10-15 drops usually suffices, even for the 10-13% range of silicon. If necessary, add a little more nitric acid as well. Evaporate to fumes of sulphur trioxide, cool slightly, dilute with water, and if necessary, boil to obtain a clear solution, cool, transfer into a measuring flask and make up to 100 ml.

For cobalt contents up to 2%, pipette 10 ml. into a 125 ml. Phillips' beaker, add 10 ml. of sodium acetate solution and 10 ml. of the nitroso-R solution and stand at room temperature for at least 5 minutes. Bring to the boil, ignoring the precipitation of phosphates. Add 5 ml. concentrated nitric acid and boil for 1½ minutes ( $\pm$  30 seconds), cool and make up to 100 ml. in a measuring flask.

Measure the light absorption on the Spekker absorptiometer,\* in a 1-cm. cell for contents of 0.5-2% cobalt and a 4-cm. cell for lower cobalt contents, using a tungsten filament lamp and Ilford Spectrum 602 filters. A "water-to-water" setting of 1.30 is convenient.

For contents in excess of 2% (contents over 6% were not investigated in these experiments) take suitable smaller aliquots and make them up to 10 ml. by adding a solution of an alloy free from cobalt, to keep the final concentrations of aluminium, salt and acids constant. Use the 1-cm. cell and the appropriate calibration graph, making allowance for the smaller aliquot taken.

#### CALIBRATION

Carry samples of a suitable cobalt-free alloy through the process to the stage of making up to 100 ml. after the fuming. To 10-ml. aliquots, add suitable quantities of the cobalt calibration solution (see below) and develop the colour as described above.

For the selection of a suitable alloy, see below under "Interferences." Theoretically, an alloy of a composition similar to the alloy to be analysed would seem

<sup>1</sup> For surveys see, for example, Sandell, E. B., "Colorimetric Determination of Trace of Metals," Interscience Publishers, 1950, 274-90 and Sandell, E. B., "Colorimetric Methods of Analysis," D. van Nostrand Co., 1949, Vol. 2, 361-77.

<sup>2</sup> "Chemical Analysis of Aluminum," 3rd Ed., 1950, 52, Aluminum Research Laboratories, New Kensington, Pennsylvania.

<sup>3</sup> Haywood, F. W. and Wood, A. A. R. (a) *J. Soc. Chem. Ind.*, 1943, **42**, 37; (b) "Metallurgical Analysis by means of the Spekker Photo-Electric Absorptiometer," Adam Hilger, Ltd., London, N.W.1, 1944, p. 47.

<sup>4</sup> Vogel, A. I., "Textbook of Quantitative Inorganic Analysis," Longmans Green & Co., Ltd., 1942, 324.

\* Any other suitable photometer can probably also be used, possibly with minor adjustments of the procedure.

desirable; practically, most alloys, or pure aluminium, can be used, as the reaction is highly specific.

The procedure outlined worked quite satisfactorily, but it seemed doubtful whether there was any point in using "Spekker acid" for aluminium alloys and, in particular, whether there was any point in driving off the nitric acid by fuming the solution so as to have only sulphuric and phosphoric acids present during the formation of the coloured complex. Whether it is permissible to have nitric acid present during the formation of the coloured complex is controversial. Some authors consider the fuming necessary,<sup>3</sup> others<sup>5</sup> state that even large quantities of nitrate ion do not interfere and still others<sup>6</sup> even work in a nitric acid medium. After the formation of metal-nitroso-R-complexes, nitric acid is added by practically all authors to decompose complexes other than that with cobalt.

This question seems of considerable practical interest, not only because of the time which could be saved if the evaporation to fumes could be omitted, but also with a view to carrying out the test for cobalt on the solution, aliquots of which serve for the photometric determination of copper, iron and nickel<sup>7</sup> in the routine of this laboratory.

It would, obviously, be very advantageous if the cobalt determination could be fitted into this and other<sup>8, 9</sup> composite schemes of analysis, all of which use nitric acid. This was indeed proved possible as the present authors found no interference by nitric acid; the following procedure was therefore evolved, which has the further advantage that alloys of all the usual ranges of silicon content can be dealt with without any modification.

### Final Method

#### SOLUTIONS REQUIRED

- (1) *Sodium Hydroxide Solution.* Dissolve 150 g. of the solid (A.R. quality) in 1 litre of water.
- (2) *Nitric Acid 1 : 1.* Concentrated acid (A.R. grade—S.G.1.42) diluted with an equal volume of water.
- (3) *Sodium Acetate Solution.* As detailed above.
- (4) *Standard Cobalt Solutions.* As described above.
- (5) *Nitroso-R Salt Solution.* 0.5% aqueous solution.

Protect from strong sunlight and make up fresh about weekly.

#### PROCEDURE

Attack a 200-mg. sample in a 250-ml. beaker with 20 ml. of the sodium hydroxide solution. Cover with a watch glass. When the violent reaction is completed, wash down sparingly, cover again and complete the reaction by boiling moderately for not more than 5 minutes.<sup>\*\*</sup> Add 20 ml. of the dilute nitric acid,<sup>9</sup> cover and heat gently until a clear solution is obtained, then boil briskly, without watchglass, for 2 minutes to

<sup>5</sup> Marston, H. R. and Lewey, D. W., *Austral. J. Exp. Biol. and Med. Science*, 1940, **18**, 343.

<sup>6</sup> Am. Soc. Testing Materials, "Tentative Photometric Method for Determination of Cobalt in Nickel," A.S.T.M. designation E 39-49 T, issued 1949.

<sup>7</sup> (a) Strosa, W., *Metallurgia*, 1945, **32**, 257; (b) "Modern Methods for the Analysis of Aluminium Alloys," by a Committee of Chemists convened by ALAR, Chapman & Hall, Ltd., 1949, pp. 70, 86, 90, 93, 139.

<sup>8</sup> Vaughan, E. J., "The Use of the Spekker Photoabsorptiometer in Metallurgical Analysis," Institute of Chemistry, London, 2 parts, 1941 and 1942.

<sup>9</sup> Unnecessary boiling takes solution silica from the glass. This may cause trouble later, particularly in the determination of copper and nickel which may be carried out from the same sample.

Allow samples with more than approximately 5% of silicon to stand on the edge of the hotplate, at moderate temperature, well below the boiling point, for 20 minutes, prior to boiling. This helps in obtaining clear final solutions on high silicon alloys; the risk of turbidity is greater for the determination of copper and nickel than for that of iron or cobalt.

<sup>10</sup> For batch work on a larger scale, dispensing devices for the alkali and acid, as described previously (Stross, W., *Analyst*, 1944, **69**, 45) can be used with great advantage.

expel nitrous fumes. It is advantageous to insert a boiling rod<sup>10</sup> at this stage to ensure rapid and smooth boiling. The boiling time should be kept reasonably uniform to ensure uniform acidity of the solutions. Transfer through a medium texture filter (e.g. Whatman No. 40) into a 200-ml. measuring flask, wash, cool to room temperature if necessary, make up to the mark and mix. Up to this point, the procedure is practically identical with that described previously for the determination of copper, nickel and iron<sup>7a, b</sup> and aliquots of this solution can, therefore, be used for these three determinations. For the determination of cobalt, pipette a 20-ml. aliquot of samples with not more than 2% of cobalt, into a 100-ml. Pyrex measuring flask, add 10 ml. of the sodium acetate solution, 4 ml. of the 0.5% nitroso-R solution and a few boiling stones.<sup>11</sup> Mix and stand for at least 5 minutes at room temperature. Place on a hotplate. Shortly before the boiling point is reached, aluminium hydroxide precipitates; this does not seem to matter, in contrast to the detrimental effect attributed<sup>12</sup> to the precipitation of beryllium hydroxide, as regards the formation of the cobalt-nitroso-R complex.

As soon as the solution is definitely boiling, cautiously add 5 ml. of concentrated nitric acid (S.G.1.42) from a burette or pipette,<sup>13</sup> without removing the flask from the hotplate. If the acid addition is delayed, the solution tends to boil up and foam rather violently, causing losses by overflowing. The first drops of the acid control the foaming instantly. Boiling then continues without interruption and is very smooth. Boil for 90 (± approximately 10) seconds and, without delay, immerse in cold water, placing a lead ring around the neck of the flask so that it can be submerged to well above the level of the liquid. The boiling time is less critical than reports in the literature,<sup>1, 13</sup> and the above description seem to imply, but in the experience of the present authors uniformity of procedure improves the consistency of results. It is quite easy to reconcile this with speedy manipulation of batches of samples, e.g. by keeping a number of flasks on the edge of the hotplate and taking only one or two at a time to the hot part of the hotplate; with a little practice they start to boil at quite regular intervals of, say, 30 seconds, so that the timing becomes quite easy.

When cooled to room temperature, make up to the mark and mix. Measure the light absorption in the same way as described for the tentative procedure.

#### Calibration Graph

In the same way, prepare a solution of a cobalt-free alloy of suitable composition (or of pure or super-pure aluminium) up to the stage of making up to 200 ml. Pipette 20-ml. aliquots into a number of 100-ml. Pyrex measuring flasks. One serves as "blank"; to the others add suitable quantities of cobalt calibration solution—e.g. 0.5, 1, 2, 4 and 8 ml., corresponding to 25–400 µg. of cobalt, equivalent to 0.125–2% of cobalt

<sup>10</sup> If no Phillip's flasks of Pyrex or similarly heat-resisting glass are to hand, use a Phillip's beaker or conical flask of similar size and transfer, after the final boiling, into an ordinary 100-ml. measuring flask.

<sup>11</sup> Stross, W., *Metallurgia*, 1951, **43**, 146.

<sup>12</sup> Carborundum pieces as recommended by Fraser, D. and Baker, R. S., *Anal. Chem.*, 1948, **20**, 1124, are very suitable. They are obtainable in this country under the designation "Carborundum grit," No. 12 or 14 being suitable sizes from the Carborundum Co. Ltd., Trafford Park, Manchester. Boil, say, a handful with aqua regia, followed by washing with water until free from acid, then dry and store in a bottle.

<sup>13</sup> Overton, T. C. J. and Parker, C. A., *Anal. Chim. Acta*, 1950, **4**, 142.

<sup>14</sup> Pipettes fitted with a rubber ball, particularly those from the "B.T.L. Pipette Dropping Bottle" (Baird and Tatlock, London, Cat. No. ZSD 3763,) are very suitable.

<sup>15</sup> McNaught, K. J., *Analyst*, 1942, **67**, 97.

in a 20-mg. aliquot of alloy. Develop the colour and measure the light absorption as described. Typical calibration graphs for 1- and 4-cm. cells are shown in Fig. 1.

The colour is fairly stable and the temperature at which the light absorption is measured is not critical within at least  $\pm 5^\circ\text{C}$ ; strong sunlight is reported to cause fading. Reasonable uniformity of timing is recommended.

### Discussion and Notes

#### (1) REPRODUCIBILITY, ACCURACY AND RANGE

A few experiments were carried out on cobalt hardeners and alloys containing cobalt, the majority on cobalt-free alloys with the addition of cobalt standard solution before the attack and at later stages. Other metals were also added in excess of normal alloying quantities (see below, under "Interferences"), either as salt solutions, or by taking samples mixed from ordinary alloys and various hardeners, e.g. of chromium, manganese, titanium and silicon. With the few exceptions mentioned below under "Interferences" the recoveries were consistently satisfactory. Nearly always the observed absorptiometer readings did not differ from those expected from the calibration graph by more than one division of the measuring drum. It is, therefore, not thought necessary to give detailed data of recoveries. Calibration graphs were prepared in parallel by the tentative and the finally adopted procedures using pure and super-pure aluminium, as well as various usual alloys. The various graphs did not differ from each other by substantially more than this amount, and the same applied to parallel determinations by the two procedures on various samples.

As shown in the calibration graph Fig. 1, 10  $\mu\text{g}$ . of cobalt in 100 ml. final solution are still easily measurable; on the 20 mg. sample represented this corresponds to 0.05% of cobalt. The sample size can be considerably increased and the range extended to lower limits, e.g. by the following alterations.

Take (a) 400 mg. of sample and attack with 20 ml. of sodium hydroxide solution and 20 ml. of nitric acid (1:1), or (b) 800 mg. of sample and attack with 40 ml. of sodium hydroxide solution and 40 ml. of nitric acid (1:1), or (c) a 1.6 g. sample and attack with 40 ml. sodium hydroxide solution and 50 ml. nitric acid (1:1). All these are made up to 200 ml. and a 20-ml. aliquot is taken. In case (b), 15 ml. of sodium acetate are added, otherwise the procedure remains unchanged, so that in the final solution 40, 80 or 160 mg. respectively are represented. The calibration graph obtained under those conditions, using pure aluminium, remains identical with that by the normal procedure. Allowance for the larger sample will, of course, have to be made in the calculation and in the question of interferences, which problem was not separately studied on the larger samples. Under conditions (c) 0.005% of cobalt is the approximate lower limit.

The upper range was tested up to just below 6%, but there is no reason to assume that the range cannot be extended further.

#### (2) CHOICE OF LIGHT FILTERS

From the reported absorption curves<sup>14</sup> of "blank" and cobalt-containing solutions, and of the Ilford Spectrum filter series<sup>3b, 7b</sup> it was to be expected that the 603, 604 and 605 filters would give rapidly decreasing

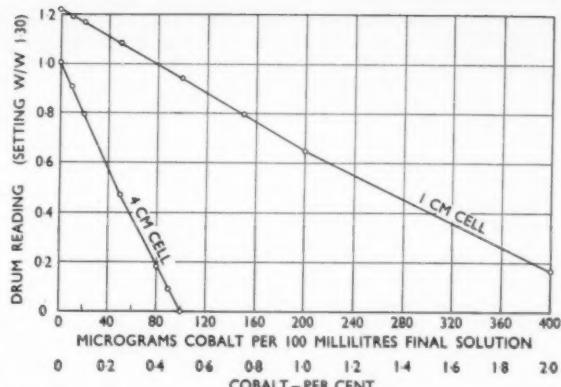


Fig. 1.—Calibration graphs for 1 cm. and 4 cm. cells using the procedure finally adopted (Ilford Spectrum filter 602 and tungsten filament lamp).

extinction values for the blanks, but a much smaller decrease of the extinction of the cobalt-nitroso-R-complex. With the filters 602 to 605 the blank extinctions decreased in the ratio 1:0.47:0.18:0.05 whilst the decrease for the cobalt complex was only in the ratio 1:0.91:0.88:0.58.\*

We think that the 602 filter, as suggested by Haywood and Wood<sup>3</sup> remains the first choice, contrary to the opinion of other authors, who recommend a wavelength corresponding to the 605 filter.<sup>14</sup> The blank value is quite manageable, even with a 4-cm. cell (and even with slightly larger addition of nitroso-R); it can, if desired, be largely eliminated by the use of an asymmetrical filter,<sup>15, 16</sup> e.g. by inserting one or two Chance OB2 filters on the left-hand side only. Only in cases when it would be desirable to increase very substantially the amount of nitroso-R (see below "Interferences") or to extend the upper limit of the measuring range without using smaller aliquots, would the choice of one of the other filters recommend itself.

#### (3) INTERFERENCE OF COPPER

Whilst some authors always remove copper, e.g. by hydrogen sulphide or electrolysis,<sup>17</sup> others state<sup>18</sup> that 12 mg. can be present in 100 ml. of the final solution; under the conditions outlined above this would be equivalent to 60% of copper.

The interfering effect of copper has been explained<sup>5</sup> as a competition between cobalt and copper for the reagent at the stage of the complex formation, i.e. at a pH greater than 2.5. Incomplete colour development is, therefore, found eventually, after the decomposition of the copper complex by nitric acid, if the amount of nitroso-R salt is not sufficient for both the cobalt and "competitors." Cobalt requires 3, copper and other metals only 1 molecule of reagent per atom of metal, but there are also influences upon the rate of complex formation which depend more critically than in the absence of interfering elements upon factors such as pH and temperature. In view of these rather complicated relations, it seemed better to obtain a survey of the practically important combinations than to attempt an exhaustive

\* By the extinction of the cobalt-complex is meant the difference between the extinctions of the blank and of the cobalt-containing solutions.

15 Stross, W., *Analyst*, 1942, **67**, 318.

16 Stross, W., *Metallurgia*, 1949, **39**, 160.

17 Young, R. S., Pinkney, E. T. and Dick, R., *Ind. Eng. Chem., Anal. Ed.*, 1946,

**18**, 474.

18 Hall, A. J. and Young, R. S., *Anal. Chem.*, 1950, **22**, 497.

investigation. The following experiments cover the ground incompletely and may have to be extended with varying requirements. Only a few experiments were made with substantially increased quantities of reagent (up to three times the standard quantity). The amount of nitroso-R salt can only be increased within comparatively narrow limits unless the excess of reagent is finally destroyed, e.g. by bromination, which is an undesirable complication of the procedure. Whilst there is no doubt that the limit of interference can be considerably influenced by increasing the amount of nitroso-R salt, this effect does not seem to increase proportionally.

It was found that with 50  $\mu\text{g}$ . of cobalt in 100 ml. of final solution, up to 8 mg. of copper did not interfere, whilst 10 mg. showed a decrease of the light absorption equivalent to an apparent "loss" of 9  $\mu\text{g}$ . of cobalt. This corresponds to an alloy with 0.25% of cobalt and 40% of copper as the approximate limit of non-interference.

For 400  $\mu\text{g}$ . of cobalt, 3 mg. of copper did not interfere, whilst 4 mg. showed an interference equivalent to an apparent "loss" of 20  $\mu\text{g}$ . of cobalt. This corresponds to an alloy with 2% of cobalt, in which case 15% of copper is the approximate limit of non-interference. Apart from increasing the amount of nitroso-R salt (which, of course, necessitates a separate calibration graph) the limit can probably also be increased by taking a smaller aliquot of alloy, but in extreme cases it will be more convenient to separate the copper, e.g. by electrolysis, which does not lead to losses of cobalt in the medium usually applied to aluminium alloys.

#### (4) NON-INTERFERENCE OF METALS OTHER THAN COPPER

The figures reported below do not indicate *limits* of non-interference, but simply the highest concentrations tested by the authors for interference and found *not* to interfere. As these concentrations were deemed to be satisfactory for practical purposes, the experiments were not extended and no conclusion is possible whether there is any interference at all. Figures obtained by the tentative procedure are marked with an asterisk, and if they differ from those given for the finally adopted method, no further conclusions can be drawn, as the difference simply means that experiments were not carried out up to the same highest concentration by both methods. So far there is no indication that the two methods differ with respect to interference.

The figures of non-interfering percentages always refer to the alloy represented in the solution (20 mg. per 100 ml. of final solution). No interference was found from Bi 5%, Cd 5%, Cr 2.8%, Fe<sup>++</sup> 5%, Fe<sup>+++</sup> 10%, Mg 15%, Mg 20%, Mn 3%, Mn 25%, Ni 5%, Ni 20%, Pb 10%, Sb 1.5%, Sb 1.5%, Si 20%, Si 12%, Sn 1%, Sn 5%, Ti 5%, Ti 5%, Zn 20%, Zn 10%.

With quantities of tin in excess of 0.5%, a turbidity of metastannic acid forms in the finally adopted technique after the addition of the nitric acid. If this is filtered off, before or after making up the solution to 200 ml., a small loss of cobalt is found. This can be avoided by adding, after the caustic soda attack, 4 ml. of concentrated hydrochloric acid before the nitric acid. This addition of hydrochloric acid has no influence on the determination of cobalt (nor on the determination of copper or nickel if carried out on aliquots of the same solution) and prevents the formation of metastannic acid from at least 5% of tin.

A number of combinations of several of the above additions were also tested for interference, usually on somewhat smaller percentages. No interference was found by, e.g. 4% Cu + 2.5% Cr + 0.25% Sb, or (tentative method) by 15% Cu + 15% Mg + 5% Fe + 3% Mn + 20% Zn + 1% Sn or (final method) by 10% Cu + 10% Fe + 10% Zn + 10% Ni, and others.

#### (5) INTERFERENCE OF NITRITE

Interference by nitrous gases has been reported in the literature<sup>14</sup> and it is recommended to boil the nitric acid before use; this precaution was found unnecessary just as the far-reaching precautions postulated by other authors<sup>5</sup>—rather more in order to avoid contamination by cobalt—of redistilling all acids and the water used from hard glass apparatus. The desire to avoid nitrite interference may also be the reason for the frequent recommendation of evaporating the metal solutions to fumes of sulphur trioxide, for which no other reason becomes clear.

Sodium nitrite solution was added before and after the sodium acetate and together with the final addition of nitric acid. 10 mg. of sodium nitrite per 20 mg. of metal had no apparent effect, whilst 50 mg. produced a small but definite decrease of the light absorption. The nitric acid used in these experiments was tested with potassium iodide and with N/56 potassium permanganate solution. The liberated iodine and the permanganate consumption indicated a nitrite content equivalent to only a few micrograms of nitrite in the appropriate quantities. Nitrous gases are, of course, also formed from metals insoluble in caustic soda after adding the nitric acid. Restricting the heating, when redissolving the precipitated aluminium hydroxide, to the barest minimum, in order to make the conditions as unfavourable as possible, the permanganate consumption indicated only up to 0.3 mg. of nitrite per 20 mg. of an alloy containing 4% of copper, but after boiling for 2 minutes this was reduced to well under 0.1 mg. No difference was found between the results of solutions boiled for times varying from just under 1 minute to 8 minutes.

Whilst normally no interference from nitrite need therefore be expected in the recommended method, it may be advisable to test the nitric acid used from time to time for nitrite content, and to purify it by boiling if necessary.

#### (6) OTHER EXPERIMENTAL CONDITIONS

(a) The strengths of the sodium hydroxide solution, the dilute nitric acid, and the sodium acetate solution are insufficiently critical to necessitate standardisation but as wide variations of these factors are not without influence, they should be kept reasonably uniform.

(b) It seemed tempting to use a boiling water bath after the nitroso-R salt and the nitric acid addition, as suggested,<sup>12</sup> but this was not found satisfactory under our conditions.

(c) The 100 ml. of final solution are, obviously, far above the necessary quantity if 1 cm. cells, let alone micro cells are used. A weighing of a few milligrams can thus be carried through the process without taking an aliquot and the method becomes a micro or semi-micro method.

#### Acknowledgments

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# Determination of Aluminium in Aluminium Bronzes Conforming to Specification D.T.D.197\*

By G. H. R. Gwatkin, B.Sc., A.R.I.C.†

(Communication from the British Non-Ferrous Metals Research Association)

*A method is proposed for the routine determination of aluminium in aluminium bronzes of the D.T.D.197 type with an accuracy of about  $\pm 1\%$  of the aluminium content. After removal of most interfering elements by mercury cathode electrolysis, aluminium is separated by 8-hydroxyquinoline from ammoniacal tartrate solution and determined volumetrically by the normal potassium bromate/bromide titration.*

THE demand for a rapid routine method for the determination of aluminium in aluminium bronzes with an accuracy of about  $\pm 1\%$  of the aluminium content led the B.N.F.M.R.A. to examine the procedures at present available. As a result it was decided to employ a mercury cathode separation of the major interfering elements, finishing volumetrically after precipitation of aluminium by 8-hydroxyquinoline from an ammoniacal tartrate solution. If much manganese is present an intermediate step (precipitation of aluminium hydroxide) is required for its removal. This paper, after a brief review of the alternative procedures available, gives a full description of the recommended technique, followed by the results of co-operative tests carried out to assess its accuracy and precision.

## Possible Procedures

The methods currently in use can be divided into two groups:—

(1) Those which involve preliminary separation from the other alloying elements by electrolysis with a mercury cathode.<sup>1,2</sup>

(2) Those which avoid this technique.

In the latter group, the following schemes were considered:—

(a) Separation of aluminium from copper, etc., by precipitation with ammonium benzoate from a buffered acetate solution containing hydroxylamine,<sup>3,4,5</sup> followed by precipitation of aluminium oxinate from ammoniacal tartrate solution.

(b) Separation of zinc, iron and manganese by precipitation in a cyanide medium (which maintains the copper in solution as the cupro-cyanide complex) followed by precipitation of aluminium oxinate from ammoniacal solution.<sup>6</sup>

Both methods may be completed gravimetrically after drying the precipitate, or volumetrically by dissolving the precipitate in hydrochloric acid and titrating the oxine with potassium bromate/bromide solution.

Procedure (b), due to W. T. Edwards, is known to give satisfactory results in the hands of experienced operators, and although the use of a large excess of cyanide may be regarded as exceptionable, no electrolytic apparatus is,

of course, required. Nevertheless, despite the ingenuity of the latter method, it is thought that the mercury cathode technique of separation has the advantage of simplicity and cleanliness: it was therefore incorporated in the proposed method.

Of the methods available for the determination of aluminium after mercury cathode separation, those involving potentiometric titration with sodium hydroxide<sup>7,8</sup> were rejected as excessively time-consuming and requiring specialised equipment. The double indicator procedure of C. H. Wood<sup>8,9</sup> was considered too susceptible to personal factors to be recommended as an accurate method. It was, therefore, preferred to finish the determination by precipitation with oxine from ammoniacal tartrate solution. The aluminium oxinate can then be determined gravimetrically or volumetrically as previously indicated.

## Reagents Required

### Acid Mixture:

Sulphuric acid (s.g.1.84)	150 ml.
Nitric acid (s.g.1.42)	125 ml.
Water	250 ml.

### Hydrochloric Acid:

50% by volume.

### Tartaric Acid:

30% solution.

### Ammonium Chloride:

2% solution.

### Potassium Iodide:

20% solution.

### Oxine Reagent:

Dissolve 25 g. 8-hydroxyquinoline in 60 ml. glacial acetic acid and dilute to 500 ml.

### Potassium Bromate/Bromide Solution, N/5:

Dissolve 5.568 g. potassium bromate and 24 g. potassium bromide in water and dilute to 1 litre.

### Sodium Thiosulphate, N/10 (approx.):

Dissolve 25 g. sodium thiosulphate crystals and 2 pellets of sodium hydroxide in water and dilute to 1 litre.

### Methyl Red Indicator:

0.2% solution in alcohol.

### Bromophenol Blue Indicator:

0.04% solution in alcohol.

### Starch Indicator:

Make 1 g. of soluble starch into a thin paste with water. Pour into 100 ml. of boiling water. Continue boiling for one minute and cool.

\* B.N.F.M.R.A. Report R.R.A.923P. The work described in this paper was made available to members of the B.N.F.M.R.A. in a confidential research report issued in August, 1951.

† Formerly Investigator, B.N.F.M.R.A. London, now with National Coal Board.

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5 Merner, G. W. C., and Townend, J., *Analyst*, 1951, **76**, 424.

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### Mercury.

All reagents should be of A.R. quality where possible.

### Procedure

#### SECTION I.—MERCURY CATHODE ELECTROLYSIS

Dissolve about 0.25 g. of alloy (Note 1 below) in 10 ml. acid mixture and evaporate until fumes of  $\text{SO}_3$  are evolved. Allow to cool, redissolve in water and dilute to about 100 ml. (Note 2). Place about 10 ml. of clean mercury in the beaker and using this as the cathode and a short length of platinum wire as anode pass a current of about 5 amps. through the solution for 40 minutes, stirring both the solution and the surface of the mercury. Wash down the cover glass and sides of the beaker once or twice during the course of the electrolysis. At the end of the specified time, decant the solution from the mercury into a 500 ml. beaker and wash the mercury by decantation 6 or 7 times with small amounts of water, adding the washings to the 500 ml. beaker. If necessary, reduce the volume of the solution to about 150 ml. by evaporation.

#### SECTION II.—REMOVAL OF MANGANESE

(This section may be omitted if manganese is known to be absent.)

Add about 10 g. of ammonium chloride and 3 drops of methyl red indicator to the nearly boiling solution and run in ammonium hydroxide (s.g. 0.880) from a burette until the indicator just turns yellow. Add 1-2 drops in excess and, with stirring, a small quantity of paper pulp. Allow the solution to stand on the hot plate for a few minutes to coagulate the precipitate. Filter through a Whatman 41 paper and wash beaker and precipitate with hot 2% ammonium chloride solution. Transfer both paper and precipitate to the original beaker, add 25 ml. hot 50% hydrochloric acid, break up the paper with a glass rod, and boil the mixture to ensure that all aluminium is in solution. If desired the volume may be increased by the addition of hot water.

#### SECTION III.—PRECIPITATION AND TITRATION OF ALUMINIUM OXINATE

To the hot solution, add 10 ml. 30% tartaric acid and five drops of bromophenol blue indicator. Run in concentrated ammonia from a burette until the indicator changes to blue and add a further 15 ml. in excess. Dilute the solution with hot water to about 200 ml., bring it just to the boil, and add 15 ml. oxine reagent, with stirring. Allow the precipitate to coagulate by digestion at 90°C. for 5-10 minutes, by which time the solution should be a clear yellow. Filter through a fairly loose pulp pad and wash four times with hot water to remove excess reagent. Extract surplus moisture from the pad by suction and transfer the pad and precipitate to the original beaker, washing the funnel with 50 ml. hydrochloric acid and then with hot water. Warm the solution until the precipitate dissolves, then cool it well, adjust the volume to about 200 ml. and run in slowly, with stirring, a measured excess (Note 3) of  $N/5$  potassium bromate/bromide solution (50-60 ml.). Add immediately 5 ml. 20% potassium iodide solution and back titrate with  $N/10$  sodium thiosulphate, using starch as indicator.

The sodium thiosulphate should be standardised by titration against 10 ml. of the bromate/bromide solution, acidified with 10 ml. hydrochloric acid + 5 ml. 20% potassium iodide.

The potassium bromate/bromide solution should be standardised against a synthetic alloy containing a known amount of aluminium carried through the whole procedure.

### Notes

- (1) Alternatively, dissolve 1 g. alloy in 10 ml. nitric acid and 20 ml. 50% sulphuric acid, evaporate to fumes of  $\text{SO}_3$  and electrolyse with mercury cathode in the usual way for about 90 minutes, decanting the solution and washings into a 500 ml. graduated flask. Make up to the mark, mix well, transfer 100 ml. aliquot portions to 500 ml. beakers and proceed as in Section II.
- (2) If the method is used for alloys of high silicon content, this element should be removed by filtration at this point before proceeding further.
- (3) The amount of potassium bromate/bromide solution in excess should be about 3 ml. A starch-iodide test paper may be used to confirm that excess is present.

### Interfering Elements

The procedure described should eliminate interference by all elements except zirconium, vanadium, titanium, beryllium, alkaline earth metals, cerium, uranium and thorium.

Zirconium (and presumably titanium) can be precipitated as the phosphate immediately after electrolysis; the filtrate from this precipitation is made up to a standard volume, suitable aliquot portions are removed and carried through sections II and III of the normal procedure.

### Results of Co-operative Tests

The accuracy of this method was verified by trials in the works laboratories of members of the Association. Each laboratory received and analysed a sample of an aluminium bronze of nominal composition :—

Aluminium	10.5%
Iron	5%
Nickel	5%
Manganese	less than 1.5%
Copper	Remainder

The results obtained by the co-operating laboratories are given in the table below :—

Laboratory Number	% Aluminium	Mean %	Spread %
1	10.60, 10.61, 10.62, 10.64, 10.65, 10.63, 10.65, 10.55, 10.55, 10.57, 10.57, 10.58, 10.60, 10.62, 10.64	10.61	0.10
2	10.52, 10.52, 10.49, 10.49, 10.52, 10.52, 10.42, 10.54, 10.52, 10.52, 10.49, 10.55, 10.49, 10.42, 10.52, 10.49	10.50	0.14
3	10.56, 10.49, 10.51, 10.53, 10.55, 10.55, 10.54, 10.52, 10.55, 10.56, 10.56, 10.49, 10.52, 10.56, 10.54	10.54	0.07
4	10.53, 10.52, 10.42, 10.45, 10.48, 10.42	10.47	0.11
5	10.55, 10.54, 10.47, 10.52	10.52	0.08

The aluminium content of this alloy was determined gravimetrically by the Association using a mercury cathode separation followed by precipitation of aluminium hydroxide and ignition to alumina at 1,150°C. The figures thus obtained were as follows :—

% Aluminium 10.55, 10.55, 10.54, 10.56, 10.55.  
Mean 10.55%.

These figures indicate that consistent results with the required accuracy can be obtained by the proposed method, but it must be emphasised that, as an earlier series of co-operative tests showed, standardisation by carrying synthetic alloy solutions through the entire procedure is of the first importance.

#### Acknowledgment

The author is indebted to the Director and Council of the British Non-Ferrous Metals Research Association for permission to publish this paper.

### International Welding Institute to meet in Sweden

THE International Institute of Welding is this year holding its annual meeting in Gothenburg, Sweden, from September 7th to 13th, by invitation of its Swedish member societies. The first item on the programme is a meeting of the Governing Council of the Institute on September 7th. The following four days will be devoted to meetings of the fifteen technical Commissions composed of representatives appointed by the societies belonging to the Institute.

On the morning of Friday, September 12th, visits have been arranged to shipyards and other works in Gothenburg and in the afternoon four papers on "Welding in Shipbuilding" will be presented by representatives of France, Great Britain, Sweden and the United States. The author of the British paper is Mr. H. H. Hagan.

The assembly closes with a meeting of the Governing Council on September 13th, after which those taking part will be free to join in a programme of works visits in Stockholm, Malmo and other industrial centres, organised by the Swedish member societies of the International Institute of Welding. A full programme of entertainment has been planned for the benefit of delegates, including a reception given by the Gothenburg City Council on September 8th and a banquet given by the host societies on September 11th.

Full particulars of the meeting and enrolment forms may be obtained from Mr. G. Parsloe, Secretary-General, The International Institute of Welding, 2, Buckingham Palace Gardens, London, S.W.1.

### French Metallurgical Meeting

THE Société Française de Métallurgie will hold its *Journées Métallurgiques d'Automne* in Paris from October 20th to 25th, 1952, at the Maison de la Chimie, 28 bis rue St. Dominique, Paris, 7e. The two main subjects to be discussed are :—

(1) Faults and fractures in metals during service.

(2) Methods of effecting economies in metals in short supply, including the rational use of alloys and the substitution of different varieties or qualities of alloys or methods of fabrication to ensure the best possible use of scarce elements.

A few communications on other subjects of particular interest will be allowed at some of the sessions. The programme will include communications by metallurgists from many European countries and from America.

A number of visits, including one to the new laboratories of the Institut de Recherches de la Sidérurgie, at Saint-Germain-en-Laye, will also be arranged. Other visits and a banquet will be held during the week.

British metallurgists will be most welcome at this meeting and those interested are asked to apply for further details to : Société Française de Métallurgie, 25, rue de Clichy, Paris, 9ème.

## Correspondence

### Analysis of Silver Solder

The Editor, METALLURGIA.

Dear Sir,

Following the paper by G. Norwitz (*Metallurgia*, 1951, 44, 276) on the analysis of silver solder by electro-deposition, a method developed during February and March of this year by Miss B. C. Winram (a second-year Chemistry student of this College) may be of interest. Silver, copper and cadmium are determined by electro-deposition from perchloric acid solution, at the appropriate voltages ; zinc is deposited in the usual way from sodium hydroxide solution.

A Griffin & Tatlock Electrochemical Analysis Apparatus was used in which D.C. is supplied to the electrodes by means of a variable transformer and a rectifier ; sufficient control of voltage was obtained by connecting a voltmeter between the electrodes. The platinum gauze electrodes used were copper plated prior to the deposition of cadmium or zinc.

*Procedure* : Dissolve 0.5—lg. sample in 5 ml. 1 : 1 nitric acid, and boil off nitrous fumes. Add 5 ml. perchloric acid (72%), evaporate to fuming and heat till nitrates are decomposed. Cool, dilute to 75–100 ml. and deposit silver at 1.2 volts (current 0.25 amps) with stirring.

Add a further 5 ml. perchloric acid, and deposit copper, with vigorous stirring at 2.2 volts (current 0.5–0.75 amps). When the cathode is removed, prior to drying and weighing, the voltage must be reduced to 2 before stirring is stopped—otherwise cadmium will commence to deposit.

Deposit cadmium at 3.4 volts with vigorous stirring, again reducing voltage before stirring is stopped, prior to removal, drying and weighing of the cathode.

Finally, add an excess of sodium hydroxide (any turbidity of the solution may be cleared with a little cyanide) and deposit zinc in the usual way.

*Results* : The following results for one silver solder were obtained by three operators, working independently.

	Chemical Analysis	R.C.W.	R.W.C.B.	J.E.B.
Silver % . . . . .	50.0	50.0	49.9	50.0
Copper % . . . . .	15.5	15.5	15.5	15.4
Cadmium % . . . . .	17.8	17.8	17.9	18.0
Zinc % . . . . .	16.6 (by diff.)	16.6	16.6	16.6
		99.9	99.9	100.0

Analyses are reported to three significant figures only, since beyond this, in our opinion, a higher accuracy is implied than ordinary methods are capable of yielding.

After the work has been checked in other laboratories, it is hoped to publish fuller details.

Yours faithfully,

R. W. C. BROADBANK,

Senior Lecturer in Inorganic Chemistry,

School of Chemistry,

Leicester College of Technology and Commerce.

Leicester.

April 30th, 1952.

# Equipment for Creep Testing at Temperatures up to 200°C.

By R. J. M. Payne,\* B.Sc., F.I.M.

*The time taken in adjusting the temperature and temperature uniformity of creep-test specimens, particularly at temperatures below 200°C., is greatly reduced by oil-bath heating of the test pieces. The particular equipment described enables six tests to be carried out simultaneously at one and the same temperature.*

CREEP testing is, by its very nature, a time-consuming operation and speedy results are not expected. Using conventional tensile creep-testing equipment, however, with ordinary wire-wound electric furnaces individually controlled, much time may be taken up in adjustment of the apparatus to secure a satisfactory degree of temperature uniformity along the specimen, and to ensure that the mean temperature approximates closely to the nominal. This is particularly true when tests are being made at temperatures below 200°C., as alterations to the currents flowing in the furnace windings only affect the temperature of the specimen after a considerable time. For these reasons, some fair degree of skill and considerable experience with the equipment are called for on the part of the operator, and a limit exists to the number of testing units that a single operator can look after at the same time.

Some two to three years ago, there was a call for the provision of further creep-testing equipment at the author's laboratories to permit an extension of investigatory work on magnesium-base alloys. Two types of alloy were being studied: (a) magnesium-rare earth-zirconium alloys, and (b) magnesium-lithium alloys. Although, as a result of developments in jet and turbo jet engines, interest in the former class of alloy now centres mainly upon creep performance at temperatures of the order of 300°C., there was a call for creep data in the temperature range 50°-200°C.: in so far as magnesium-lithium alloys are concerned interest was confined to creep performance at temperatures below 100°C. The equipment to be described was devised to enable tests to be carried out at temperatures not exceeding 200°C., and this object is achieved with an ease and economy in time not possible with standard equipment. Although not outstandingly novel, the design is unconventional, and it is thought that a description of the equipment may be of interest to others engaged in creep testing at these moderate temperatures.

## Description of the Equipment

The apparatus illustrated in Figs. 1 and 2 enables tests to be carried out on six specimens simultaneously at one and the same temperature. Specimens *A* are immersed directly in an oil bath *C* of 12 gallons capacity, electrically heated, and with the oil vigorously circulated by means of a propeller. So far as can be seen, there are no objections to this practice where, as in the present case, the stresses used in testing are confined to those giving creep rates which are of practical interest and importance: it is, however, conceivable that in creep-to-rupture testing there might be objection to direct contact between specimen and oil. A single temperature

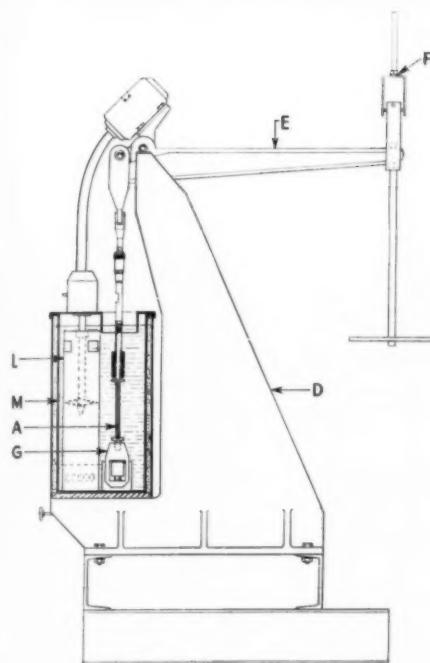


Fig. 1.—A general view of the equipment.

regulator controls the temperature of the bath, and creep specimens can be removed and fresh ones inserted without disturbing tests in progress. Test specimens of 0.564 in. diameter and 5 in. gauge length are used with this machine: much creep testing has been done, and is done, elsewhere on smaller specimens, but as our work was largely concerned with cast alloys, where the grain size may be appreciable in relation to the diameter, the larger specimen was preferred. The component parts of the machine have been designed for operation with stresses of up to 10 tons/sq. in. on a 0.564 in. diameter specimen. A stress of this magnitude calls for a dead weight loading on the end of the lever of 500 lb. In the interests of compactness, the present machine was designed with the test specimens at 6-in. centres and, to accommodate the necessary weights without fouling, alternately long and short hangers are used.

The general arrangement of the machine is shown in Fig. 2. It will be seen that three main frame castings *D* are provided, each with two test stations. The present equipment has thus six test stations, but machines with

\* Metallurgical Research Laboratory, J. Stone & Co. (Charlton), Ltd.



A Creep test specimen.  
B Extensometer.  
C Oil bath.

D Main frame.

E Loading lever.

F Unloading mechanism.

N Temperature-sensitive element for temperature controller.

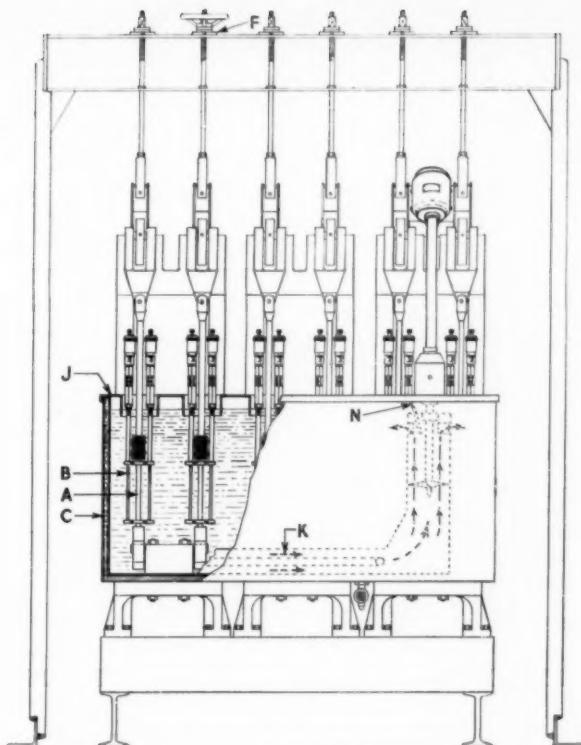


Fig. 2.—Sketch showing the details of the apparatus.

two, four, eight or more stations could be made if preferred. The main frame castings are bolted to an underframing of heavy steel sections. Loads are applied to the ends of the levers *E* which are provided with the usual hardened steel knife edges, and a screw mechanism *F*, engaging with the upper ends of the hangers, enables the load to be applied to the lever at the commencement of a test and removed afterwards without shock. The test specimen is screwed into a shackle *G* at the lower end, a  $\frac{1}{4}$ -in. diameter steel ball being interposed between the shackle and the lower anchor block *H* to ensure axial loading. It will be appreciated that when a fresh specimen has to be placed in the machine, the fitting operation has to be carried out "blind" as the oil is opaque: the specimen with extensometer *B* and top shackle attached, has therefore to be screwed into the lower shackle by touch. To help in mating the threads the shackle *G* is allowed only a limited degree of freedom, so that it is always substantially in a vertical position. The screwed socket of the shackle is, furthermore, made slightly bell-mouthed to help guide the test-piece into position, and no difficulty has been experienced in fitting a fresh test-piece.

The oil tank is of welded mild steel, lagged with glass wool and covered externally with sheet aluminium. Sindanyo board, 1 in. thick, is placed beneath the tank to restrict the loss of heat by conduction to the frame. The bolts carrying the load from the anchor blocks down to the main casting pass through the bottom of the tank

and are brazed thereto to prevent oil leaks: no stresses fall on these joints. To avoid exposing an unnecessarily large surface of oil to the atmosphere, the tank is covered with a (fixed) sheet steel lid *J*: six wells are provided to accommodate the creep specimens and extensometers.

The oil is heated by means of loops  $\frac{1}{4}$ -in.  $\times$  0.012 in. nickel-chromium alloy ribbon carried on a frame and tensioned by means of a spring. The heater is situated in a sheet steel tunnel *K*, 3 in.  $\times$  2 in.  $\times$  24 in. long, located at the bottom of the tank and connected to the vertical shaft *L* on which the propeller *M* revolves. In this way, oil is drawn from the bottom corner of the tank, passes over the whole of the heater winding and is discharged at a diagonally opposite top corner. The oil circulates rapidly and a high degree of uniformity of temperature is secured. The temperature-sensitive element *N* of the temperature controller is placed in the vertical shaft so that it is in contact with the oil immediately after it has passed over the heated winding. Power consumption when running at 200° C. is about  $2\frac{1}{2}$  kW.

For the work on which we were engaged, strain measurements could be made with sufficient accuracy using micrometers, but there is no reason why optical extensometers should not be used if desired. Micrometers with thimbles of  $1\frac{1}{2}$ -in. diameter and graduated to 0.0001 in. are employed, the extensometers being fixed to collars machined on the test-bar at the ends of the 5-in. gauge length.

Two safety devices are fitted to the equipment. Oil fires could be started by an overheating of the bath as a whole as a result of a failure of the control equipment, or by local overheating in the event of a breakdown of the oil circulating arrangements. To guard against the first contingency a trip switch containing a fusible element of pure tin (melting point 232° C.) is placed in the bath to cut off the current in the main heater circuit. A centrifugal switch mounted on the propeller shaft interrupts the main current if the propeller is not working.

### Performance

With creep-testing equipment it is required that :

- (1) The temperature of the specimen shall be sensibly uniform (at least over the gauge length) at any particular moment ;
- (2) short-term variations in the mean temperature of the test-piece resulting from the switching on and off of heaters shall be small ; and
- (3) the temperature in the hot space shall be virtually independent of room temperature changes.

These requirements are met using a platinum resistance thermometer and proportioning relay of the Prosser type. By virtue of the large mass of oil in use and the vigorous circulation, temperature differences as between specimen and specimen, and over the gauge length of a single specimen are very small indeed (less than  $\frac{1}{2}$ ° C.) at any given moment : in fact, after preliminary trials only a single thermocouple for temperature measurement was used in practical testing. Short-term variations in temperature resulting from the switching of heating currents are of the order of 0.2° C.

As would be expected from control equipment using the resistance thermometer principle, the temperature of the oil bath is virtually unaffected by changes in room temperature. Long term variations are well within the tolerance permitted in B.S.1687-1950, namely,  $\pm 1^\circ$  C., and are usually of the order of  $\frac{1}{2}$ ° C.

The apparatus has been in regular use over a period of 12 months and has given no trouble. Creep curves obtained with the machine have been indistinguishable from those obtained with conventional equipment in which the specimen is heated in air. Shell Voluta oil has been found suitable for running at 200° C., and although there is a slight smell of hot oil at this temperature, an exhaust fan removes it ; there is no appreciable smell at temperatures lower than 150° C. A less viscous oil than the Voluta is desirable for operation at temperatures lower than 100° C. and here ordinary machine oil is suitable.

The facility with which creep tests can be carried out using this equipment has made us wish that the same principles could be applied to testing at higher temperatures.

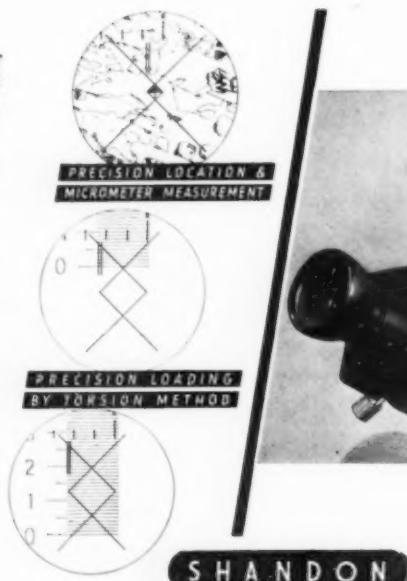
### Acknowledgments

The author is indebted to the directors of J. Stone & Co. (Charlton), Ltd. for permission to publish a description of the equipment and acknowledges with pleasure the assistance given by Mr. M. L. Franck, B.Sc., and Mr. J. Funnell in connection with matters relating to mechanical design and temperature control respectively. The equipment was built in the Laboratory workshop under the control of Mr. E. H. Sherwood.

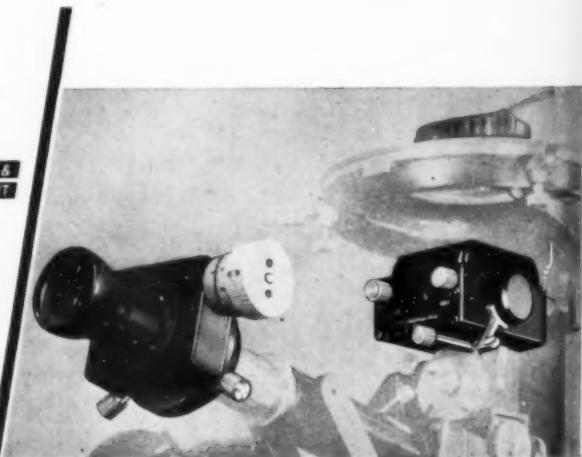
## A PRACTICAL ACCURATE INSTRUMENT FOR LOW-LOAD HARDNESS TESTS

### The REICHERT micro-hardness tester

The Reichert Micro-hardness Tester gives reliable information concerning the hardness of the constituent crystals of complex substances, or the hardness of thin sheets or coatings. The indentation can be located precisely ; loading is on the torsion principle and the load applied is simply read off on a scale which appears in the eyepiece at the touch of a lever—there are no cumbersome weights to contend with. The diagonal length of the indentation is measured by means of the special hardness testing eyepiece which incorporates an expanding square and a measuring scale coupled with a micrometer drum. We sincerely believe that this apparatus is the most advanced, practical and convenient now available for implementing the relatively new but important technique of low-load hardness testing.



SHANDON



SHANDON SCIENTIFIC COMPANY, 6, CROMWELL PLACE, LONDON, S.W.7. KNightsbridge 1131

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